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LABORATORY QUALITY CONTROL MANUAL

Robert S. Kerr Water Research Center
Ada, Oklahoma

1969

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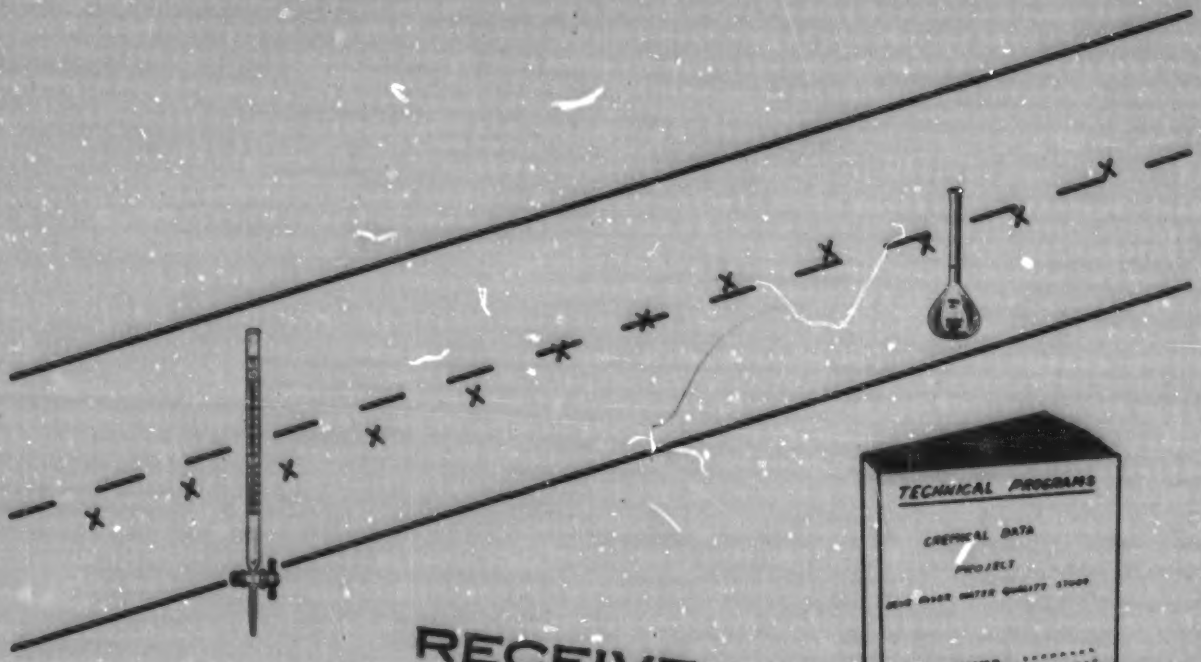

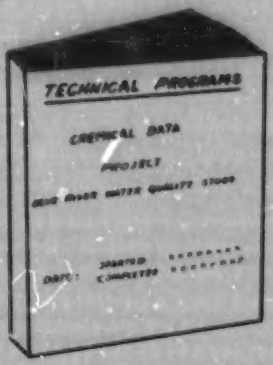

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FEDERAL WATER POLLUTION CONTROL ADMINISTRATION
SOUTH CENTRAL REGION
ROBERT S. KERR WATER RESEARCH CENTER

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FOREWORD

Those who generate water quality data have a serious responsibility to those who will use it. Such data often become the basis for various action programs in water pollution control. Among these are: (1) the construction and operation of wastewater treatment works costing millions of dollars; (2) the early detection of trends in water quality degradation that, if allowed to go unchecked, could result in the loss of beneficial water uses; and (3) court actions that could result in the levying of heavy fines and other penalties and even industrial shutdowns.

The significance of water quality data precludes any thought of careless laboratory operation; however, even the best staffed, equipped, and maintained laboratories need some measure of product quality. Conscientious personnel and well equipped laboratories are not enough.

The Federal Water Pollution Control Administration (FWPCA) is concerned about laboratory quality and has initiated a program of improved effort in that direction. This manual deals with two areas of that program; statistical analytical quality control and record keeping. Product quality control is an old technique of the manufacturing industries. The statistics underlying product quality control are a proven technique but their application to routine laboratory production is new. This manual describes statistical techniques as applied to analytical quality control. Properly understood and judiciously applied in the laboratory, they can impart the same confidence in the

reliability of water quality data as process industries have in their products.

This manual is also concerned with record keeping as it applies to laboratory procedures. Critics of water quality surveys and studies based on laboratory data are entitled to examine laboratory computations and records. It is important, then, that records and calculations be maintained in presentable and defensible form. This manual suggests a method of laboratory record keeping that should satisfy the most severe critic.

These two areas of improved laboratory operation cannot be achieved without additional laboratory supervision and workloads, but the results will show in good laboratories becoming better and laboratory personnel gaining new confidence in their products. Even more important, users of the laboratory data can confidently plan and implement the programs which the data imply.

ANALYTICAL QUALITY CONTROL LABORATORY
DIVISION OF WATER QUALITY RESEARCH
FEDERAL WATER POLLUTION CONTROL
ADMINISTRATION

The Federal Water Pollution Control Administration (FWPCA) gathers water quality data to determine compliance with water quality standards, to provide information for planning of water resources development, to determine the effectiveness of pollution abatement procedures, and to assist in research activities. In a large measure, the success of the pollution control program rests upon the reliability of the information provided by the data collection activities.

To insure the reliability of physical, chemical, and biological data, FWPCA's Division of Research has established the Analytical Quality Control (AQC) Laboratory at 1014 Broadway, Cincinnati, Ohio. The AQC program conducted by this Laboratory is designed to assure the validity and, where necessary, the legal defensibility of all water quality information collected by FWPCA.

The AQC Laboratory is responsible for:

Conducting analytical methods research, providing leadership in the selection of laboratory procedures, conducting a reference sample program for methods verification and laboratory performance, and advising laboratories in the development of internal quality control. In addition, the

Laboratory develops and evaluates automatic water quality monitoring instrumentation and assists FWPCA's nine Regions in the procurement and installation of this type of equipment.

METHODS RESEARCH

Although analytical methods are available for most of the routine measurements used in water pollution control, there is a continuing need for improvement in sensitivity, precision, accuracy, and speed. Development is required to take advantage of modern instrumentation in the water laboratory. In microbiology, the use of new bacterial indicators of pollution, including pathogens, creates a need for rapid identification and counting procedures. Biological collection methods need to be standardized to permit efficient interchange of data. The AQC Laboratory devotes its research efforts to the improvement of the routine "tools of the trade."

METHODS SELECTION

Assisted by Advisory Committees, the AQC Laboratory provides a program for selecting the best procedures in water and waste analysis from among those that are available. Through the publishing of FWPCA methods manuals, updated regularly, the program insures the application of uniform analytical methods in all laboratories of FWPCA. The validity of the chosen procedures and the evaluation of analytical performance are verified by reference sample studies involving participation by regional, basin, and project laboratory staff personnel. The

FWPCA methods manual is available to any organization upon request.

INTRALABORATORY QUALITY CONTROL

To maintain a high level of performance in daily activities, every analytical laboratory must provide a system of checks on the accuracy of reported results. While this is a basic responsibility of the analyst and his supervisor, the AQC Laboratory provides guidance in the development of model programs which can be incorporated into the laboratory routine.

AQC REGIONAL COORDINATORS

The Administration-wide quality control program is carried out through FWPCA Regional AQC Coordinators. The Coordinator, appointed by the Regional Director, implements the program in his regional laboratory and maintains relations with state and interstate pollution control agencies within the region to encourage their use of FWPCA methods and active participation in the analytical quality control effort. In addition, the Coordinator brings to the attention of the AQC Laboratory the special needs of his region in analytical methodology.

U. S. GEOLOGICAL SURVEY

Because water quality surveillance is a joint program between FWPCA, the U. S. Geological Survey and the states, the AQC Laboratory works closely with the USGS in securing uniform methods in both agencies.

Through regular interchange of procedural outlines and joint participation in reference sample studies, the two agencies seek to promote complete cooperation in water quality data acquisition.

PROFESSIONAL LIAISON

The Laboratory staff, along with other FWPCA scientists, actively participates in the preparation of *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association) and in subcommittee and task group activities of Committee D-19 of the American Society for Testing and Materials. A senior member of the AQC Laboratory staff is General Referee for Water, Subcommittee D, of the Association of Official Analytical Chemists.

For further information write your Regional Coordinator or

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Analytical Quality Control Laboratory
Federal Water Pollution Control Administration
U. S. Department of the Interior
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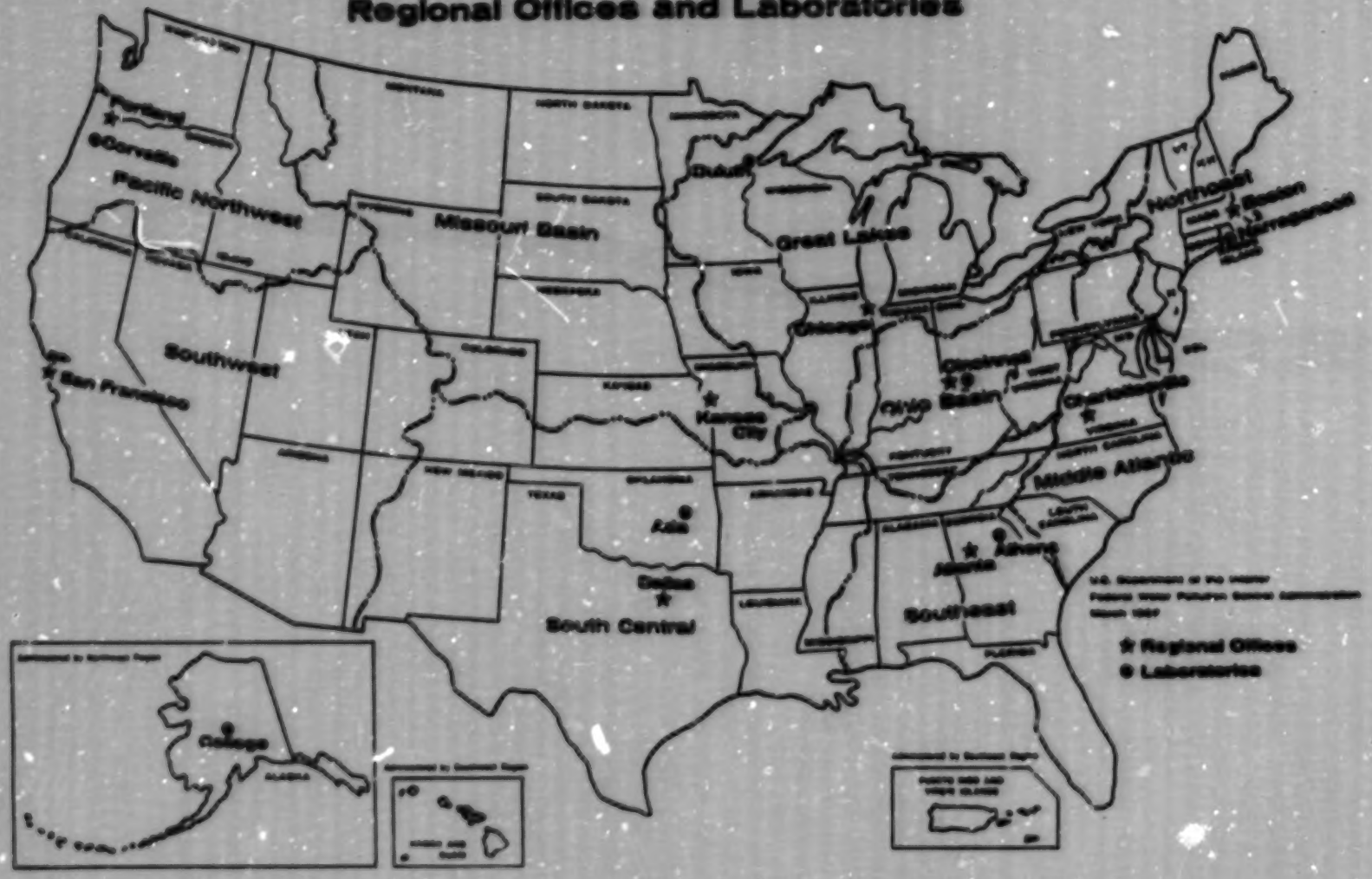
Federal Water Pollution Control Administration

Regional Offices and Laboratories



INTRODUCTION

The precision and accuracy of analytical data produced in the laboratory can be detected by using quality control charts. These control charts serve as "fingerprints" of a laboratory's operations. In addition, the use of these charts enables a supervisor to validate the data produced from a specific laboratory group for the analysis of a specific parameter. These charts indicate when the laboratory is operating normally or abnormally, thus pointing out when data generated should be accepted, questioned, or rejected. In the same manner they indicate when the laboratory is operating at optimum efficiency.

CONSTRUCTION OF CONTROL CHARTS

Two control charts are required to "fingerprint" the laboratory operations for a given analytical procedure. These are referred to as precision and accuracy control charts. Precision control charts are constructed from duplicate sample analyses, whereas accuracy control charts are constructed from spiked or standard sample analyses data. A set of these control charts represents, and is restricted to, a specific laboratory, group of analysts, analytical method, range of concentration, and period of time. To construct the precision and accuracy control charts it is recommended that at least 20 sets of duplicate and 20 sets of spiked sample data from an in-control process be used for the initial construction. The selection of in-control

data can be made on a judgment basis.

It is necessary that the initial sets of data be obtained under the following conditions:

1. Normal laboratory operations
2. Constant analyst or group of analysts
3. Consistent method
4. Narrow range of concentration of the parameter analyzed.

Since the precision and accuracy of the analyses of many parameters are proportional to the concentration of the parameter to be measured, it may be necessary to use several control charts in many different ranges of concentrations for a given parameter.

The control charts are derived from three basic calculations:

1. Standard deviation (S_d) of the differences between duplicates or, in the case of spiked or standard samples, between the known quantity and the quantity obtained.
2. The upper control limit (UL)
3. The lower control limit (LL)

Prior to these calculations, two decisions must be made:

1. The α and β levels
2. The allowable variability levels

By definition, α is the probability of judging the process to be out of control, when in fact, it is in control. It is recommended that α be chosen to lie between the boundaries of .05 and .15, that is, the laboratory personnel are willing to stop the laboratory process somewhere between 5 and 15% of the time, judging it to be out of control, when in fact, it is in control. If the cost of examining a process to determine the reason or reasons for being out of control is considerable, then it may be desirable to choose a low α. Likewise, if the cost is negligible, it may be desirable to choose a larger α value, and thus stop the process more frequently.

On the other hand, β is defined as the probability of judging the process to be in control when it is not. Again, it is recommended that β be chosen to lie between the values of .05 and .15; thus, the laboratory personnel are willing to accept out of control data somewhere between 5 and 15% of the time. The economic considerations used for choosing α are also applicable to the choice of β.

It is also essential to set maximum and minimum allowable variability levels. It is necessary to specify a value for the minimum and maximum amount of variation that will be allowable in the system. These minimum and maximum amounts are referred to as σ_0^2 and σ_1^2 respectively. The values used should be based on a knowledge of the variation in the procedure under consideration. However, if such knowledge is not available, the values may be arbitrarily set at $\sigma_0^2 = (\sigma - .20\sigma)^2$ and $\sigma_1^2 = (\sigma + .20\sigma)^2$.

Mathematical Equations

$$S_d^2 = \frac{\sum_{i=1}^{n} di^2 - \frac{(\sum_{i}^{n} di)^2}{N}}{N - 1} = \text{Variance of the differences}$$

$$S_d = \sqrt{S_d^2} = \text{Standard deviation of the differences} \qquad (1)$$

$$S_o^2 = (.8S_d)^2 = .64\, S_d^2 \text{ estimates } \sigma_o^2$$

$$S_1^2 = (1.2S_d)^2 = 1.44\, S_d^2 \text{ estimates } \sigma_1^2$$

$$UL(M) = \frac{2 \log_e\left[\frac{1-\beta}{\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} \qquad (2)$$

$$LL(M) = \frac{2 \log_e\left[\frac{\beta}{1-\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} \qquad (3)$$

Where: UL(M) = upper limit at M sets of samples

LL(M) = lower limit at M sets of samples

di = the difference between the i^{th} set of duplicates or spiked samples

N = the total number of sets of duplicates or spiked samples used to construct the control charts

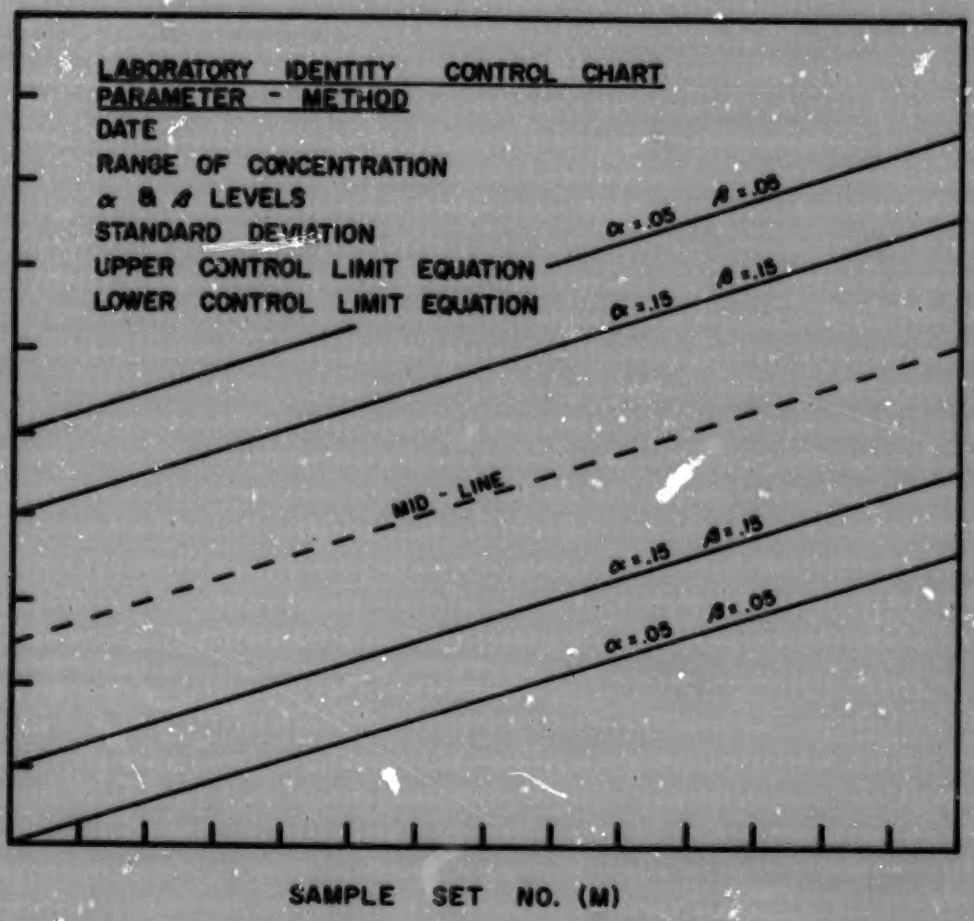
S_o^2 = minimum amount of variation allowed in the system

S_1^2 = maximum amount of variation allowed in the system

α = percent (decimal fraction) of time you are willing to judge the procedure out of control when it is in control

β = percent (decimal fraction) of time you are willing to judge the procedure in control when it is out of control

M = number of sets of duplicates or spiked samples used in calculating the value to be plotted on the chart



EFFECT OF α & β LEVELS ON
STANDARD CONTROL CHART

CONTROL CHART

CALCULATIONS

EXAMPLE I

Accuracy Control Chart

Laboratory: Laboratory A

Parameter Analyzed: Total phosphate phosphorus

Method: Colorimetric with persulfate digestion

Date: November 12, 1968

Data:

Results of Analyses of Standards

(mg/l Total PO_4-P)

Actual	Obtained	Difference (di)	di^2
.34	.33	+.01	.0001
.49	.49	.00	.0000
.49	.49	.00	.0000
.68	.65	+.03	.0009
.67	.65	+.02	.0004
.66	.70	-.04	.0016
.83	.80	+.03	.0009
.34	.34	.00	.0000
.50	.47	+.03	.0009
.40	.40	.00	.0000
.50	.53	-.03	.0009
.66	.60	+.06	.0036
.50	.56	-.06	.0036
.52	.59	-.07	.0049
.98	.75	+.23	.0529
.49	.63	-.14	.0196
1.6	1.7	-.10	.0100
1.3	1.2	+.10	.0100

Actual	Obtained	Difference (di)	di^2
3.3	3.3	.00	.0000
4.9	4.6	+.30	.0900
2.3	2.3	.00	.0000
1.3	1.3	.00	.0000
2.3	2.4	-.10	.0100

$$\Sigma di = .27$$

$$\Sigma di^2 = .21$$

$$(\Sigma di)^2 = .07$$

Calculations

$$S_d^2 = \frac{\Sigma di^2 - \frac{(\Sigma di)^2}{N}}{N-1} = \frac{.21 - \frac{.07}{23}}{22} = .009$$

$$S_d = \sqrt{S_d^2} = \sqrt{.009} = \pm .09 \qquad (1)$$

$$S_o^2 = (.8S_d)^2 = .64\, S_d^2 = .64(.009) = .006$$

$$S_1^2 = (1.2S_d)^2 = 1.44\, S_d^2 = 1.44\,(.009) = .013$$

$$UL(N) = \frac{2\log_e\left[\frac{1-\beta}{\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + N\,\frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$= \frac{3.5}{\frac{1}{.006} - \frac{1}{.013}} + M \frac{\log_e \frac{.013}{.006}}{\frac{1}{.006} - \frac{1}{.013}}$$

$$= \frac{3.5}{90} + M \frac{.69}{90}$$

$$= .039 + .0077(M) \qquad (2)$$

$$LL(M) = \frac{2 \log_e \left[\frac{\beta}{1-\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e \left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$= \frac{-3.5}{90} + M \frac{.69}{90}$$

$$= -.039 + .0077(M) \qquad (3)$$

Upper limits on the Y-axis:

at M = 0

UL (0) = .04 + 0(.008) = .04;

at M = 14

UL (14) = .04 + 14(.008) = .15

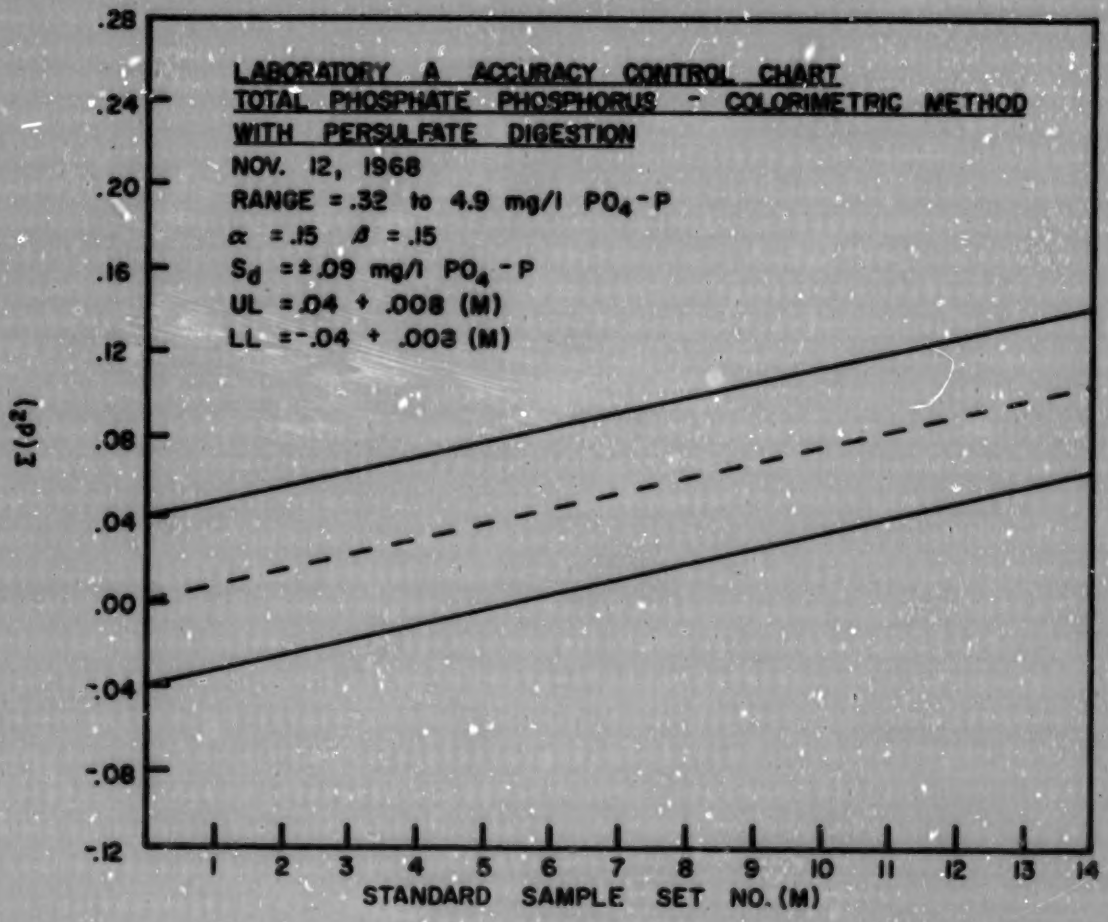
Lower limits on the Y-axis:

at M = 0

LL (0) = -.04 + 0(.008) = -.04;

at M = 14

LL (14) = -.04 + 14(.008) = .07



EXAMPLE i - ACCURACY CONTROL CHART

EXAMPLE II

Precision Control Chart

Laboratory: Laboratory AC

Parameter Analyzed: Hexane extractables

Method: Semiwet extraction method

Date: January 5, 1969

Data:

Results of Analyses of Duplicate Samples

(mg/l Hexane Extractables)

Duplicate No. 1	Duplicate No. 2	Difference (di)	di^2
.40	.50	+.10	.0100
.80	.83	+.03	.0009
.63	.60	-.03	.0009
.93	.83	-.10	.0100
1.46	1.16	-.30	.0900
1.20	1.10	-.10	.0100
1.80	1.56	-.24	.0576
2.16	2.20	+.04	.0016
.40	.36	-.04	.0016
.20	.28	+.08	.0064
.40	.30	-.10	.0100
.46	.40	-.06	.0036
.40	.60	+.20	.0400
1.76	1.80	+.04	.0016
.83	.86	+.03	.0009
1.16	1.02	-.14	.0196
.56	.63	+.07	.0049
1.26	1.33	+.07	.0049

Duplicate No. 1	Duplicate No. 2	Difference (di)	di^2
.48	.36	-.12	.0144
.59	.59	.00	.00
.59	.60	+.01	.0001
1.17	1.26	+.09	.0081

$\Sigma di = -.470$

$\Sigma di^2 = .297$

$(\Sigma di)^2 = .221$

Calculations

$$S_d^2 = \frac{\Sigma di^2 - \frac{(\Sigma di)^2}{N}}{N-1} = \frac{.297 - \frac{.221}{22}}{21} = .0137$$

$$S_d = \sqrt{S_d^2} = \sqrt{.0137} = .117 \qquad (1)$$

$$S_o^2 = (.8S_d)^2 = .64\, S_d^2 = .64(.0137) = .00877$$

$$S_1^2 = (1.25_d)^2 = 1.44\, S_d^2 = 1.44(.0137) = .01973$$

$$UL(M) = \frac{2 \log_e \left[\frac{1-\beta}{\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e \left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$= \frac{3.5}{\frac{1}{.0088} - \frac{1}{.0197}} + M \frac{\log_e\left[\frac{.0197}{.0088}\right]}{\frac{1}{.0088} - \frac{1}{.0197}}$$

$$= \frac{3.47}{63.35} + M \frac{.811}{63.35}$$

$$= .054 + .0128(M) \qquad (2)$$

$$LL(M) = \frac{2 \log_e\left[\frac{\beta}{1-\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$= \frac{-3.47}{63.35} + M \frac{.811}{63.35}$$

$$= -.054 + .0128(M) \qquad (3)$$

Upper limits on the Y-axis:

at M = 0

UL (0) = .05 + 0(.013) = .05;

at M = 14

UL (14) = .05 + 14(.013) = .23

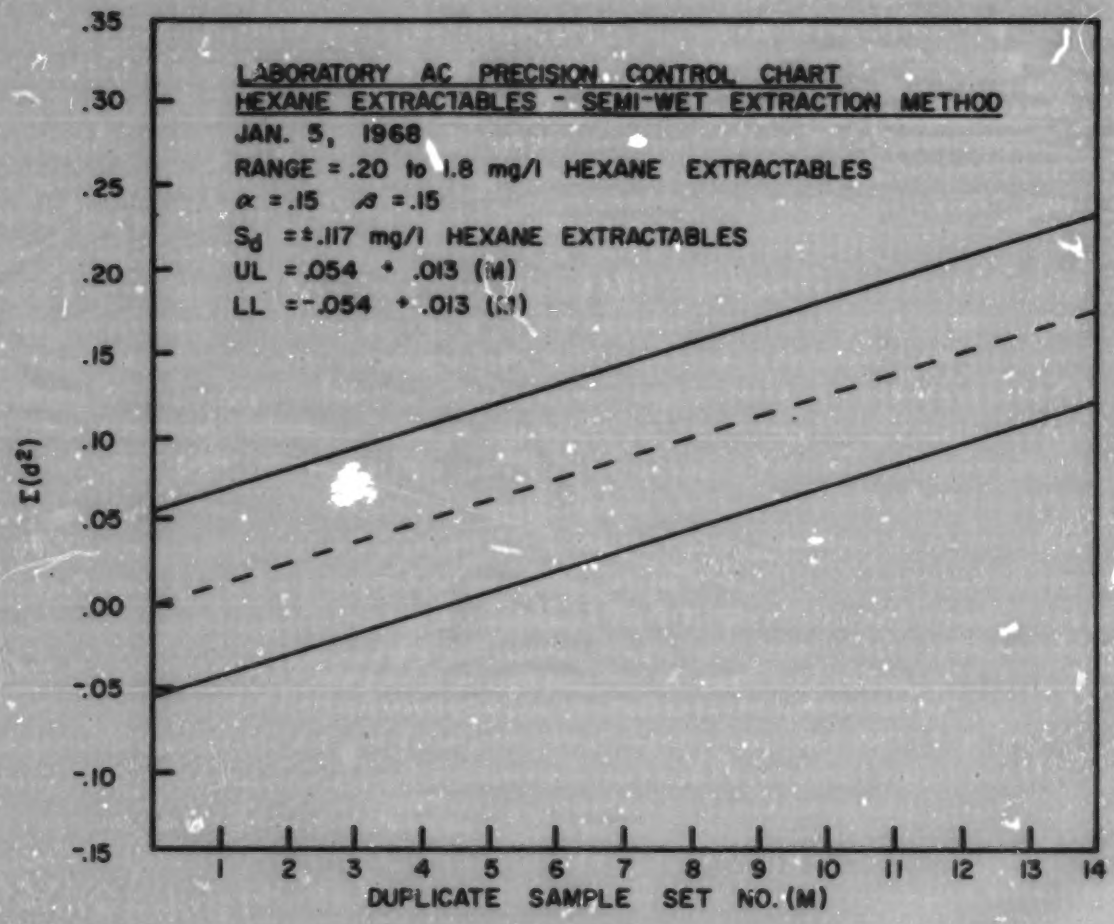
Lower limits on the Y-axis:

at M = 0

LL (0) = -.05 + 0(.013) = -.05;

at M = 14

LL (14) = -.05 + 14(.013) = .13



EXAMPLE 2 - PRECISION CONTROL CHART

USE OF CONTROL CHARTS

Once the control charts are constructed, and prior to their use, consideration must be given to the number of duplicate analyses to be conducted during a series of samples. Likewise, the same decision must be made on spiked or standard samples.

In considering the number of duplicate and spiked sample analyses to be conducted in a series of samples, it is necessary to weigh the consequences when the data go out of control. The consequences of this situation are reanalyzing a series of samples or discarding the questionable data obtained. The samples to be reanalyzed are those lying between the last in-control point and the present out-of-control point. A realistic frequency for running duplicate and spiked samples would be every fifth sample; however, economic consideration and experience may require more or less frequent duplicate and spiked sample analyses.

Once the frequency of duplicate and spiked samples has been determined, it is then necessary to prepare spiked or standard samples in concentrations relative to the concentration of the control charts, which should be similar to those of the environmental samples. These spiked or standard samples must be intermittently dispersed among the series of samples to be analyzed and without the analyst's knowledge of concentration. Similarly, duplicate samples must be intermittently dispersed throughout the series of samples to be analyzed, and ideally, without the analyst's knowledge; however, this is sometimes very difficult

to accomplish.

The results of the duplicate and spiked sample analyses should be calculated immediately upon analyzing the samples to allow for early detection of problems that may exist in the laboratory. An example of these calculations follows:

Duplicate Sample No. M	Results No. 1	Results No. 2	Difference (di)	di^2	$\Sigma(di^2)$
1	5.4	5.2	.2	.04	.04
2	4.8	4.7	.1	.01	.05
3	6.1	5.8	.3	.09	.14

Upon plotting the summation or $\Sigma(di^2)$, one of three possibilities can occur:

1. Out of control on the upper limit
2. In control within the upper and lower limit lines
3. Out of control on the lower limit

Out of Control on Upper Limit

When data goes out of control on the upper limit the following steps should be taken:

1. Stop work immediately
2. Determine problems
   a. Precision control chart
      (1) The analyst

(2) Nature of the sample

(3) Glassware contamination

b. Accuracy control chart

(1) The analyst

(2) Glassware contamination

(3) Contaminated reagents

(4) Instrument problems

(5) Sample interference with the spiked material

3. Rerun samples represented by that sample set number, including additional duplicate and spiked samples.

4. Begin plotting at sample No. 1 on chart.

In Control

When data continuously fall in between the upper and lower control limits, the analyses should be continued until an out-of-control trend is detected.

Out of Control on Lower Limit

When data fall out of control on the lower limit, the following steps should be taken:

1. Continue analyses unless trend changes

2. Construct new control charts on recent data

3. Check analyst's reporting of data.

The primary reasons for data falling out of control on the lower limit are increased efficiency or false data reporting.

Standard Deviation

The purpose of calculating the standard deviation is to allow for inter-laboratory comparisons of precision and accuracy as well as similar comparisons with the literature. In this respect the standard deviation can be used as a guide to determine if the laboratory is operating "in the ball park" on precision and accuracy for a given parameter. It should be emphasized that the comparisons of standard deviations should only be used as a guide since the standard deviation of a specific laboratory is characteristic of that laboratory's operations and no other.

OUTLINE OF PROCEDURES

FOR

CONSTRUCTING AND USING CONTROL CHARTS IN THE LABORATORY

1. Obtain initial sets of duplicate and spiked or standard sample data for a given parameter under the following conditions -
   a. Normal laboratory operations
   b. Constant analyst or group of analysts
   c. Consistent method
   d. Parameter present in a narrow range of concentration
2. Calculate the following -
   a. Standard deviation
   b. Upper control limits
   c. Lower control limits
3. Construct control charts for precision and accuracy. These charts represent and are restricted to the specific -
   a. Laboratory
   b. Parameter
   c. Range of concentration
   d. Analytical method
   e. Time
4. Use of control charts -
   a. Analyze duplicate samples intermittently throughout day's samples

b. Analyze spiked or standard samples intermittently dispersed among day's samples without analyst's knowledge of concentration.

c. Calculate $\Sigma(di^2)$ of results as soon as possible

d. Plot $\Sigma(di^2)$ -

(1) Out of control on upper limit -

(a) Stop work

(b) Determine problems

(c) Rerun samples represented by that number

(d) Begin plotting at sample No. 1

(2) In control - continue analyses

(3) Out of control on lower limit -

(a) Continue analyses unless trend changes

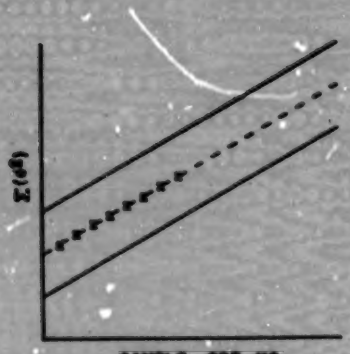
(b) Construct new chart on recent data

(c) Check analyst reporting data

e. Compare standard deviation

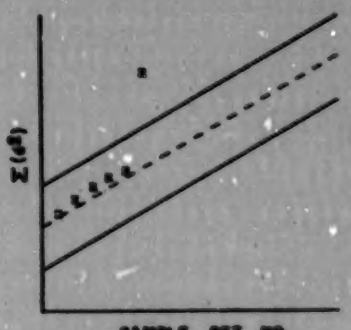
(1) Other laboratories

(2) Literature



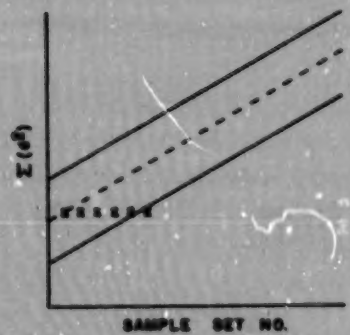
ANALYSIS IN CONTROL

NO PROBLEMS:
CONTINUE ANALYSIS



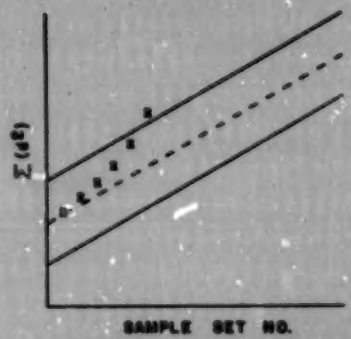
ANALYSIS OUT OF CONTROL
UPPER LIMIT

PROCEDURES:
1. STOP ANALYSIS
2. LOCATE PROBLEM
3. CORRECT PROBLEM
4. RERUN SAMPLES
5. START CHART AT SAMPLE SET NO. 1.



ANALYSIS OUT OF CONTROL
LOWER LIMIT

INCREASED EFFICIENCY OR
FALSE REPORTING
PROCEDURES:
1. CONTINUE ANALYSIS
2. CONSTRUCT NEW CHART
WITH RECENT DATA
3. OBSERVE ANALYST

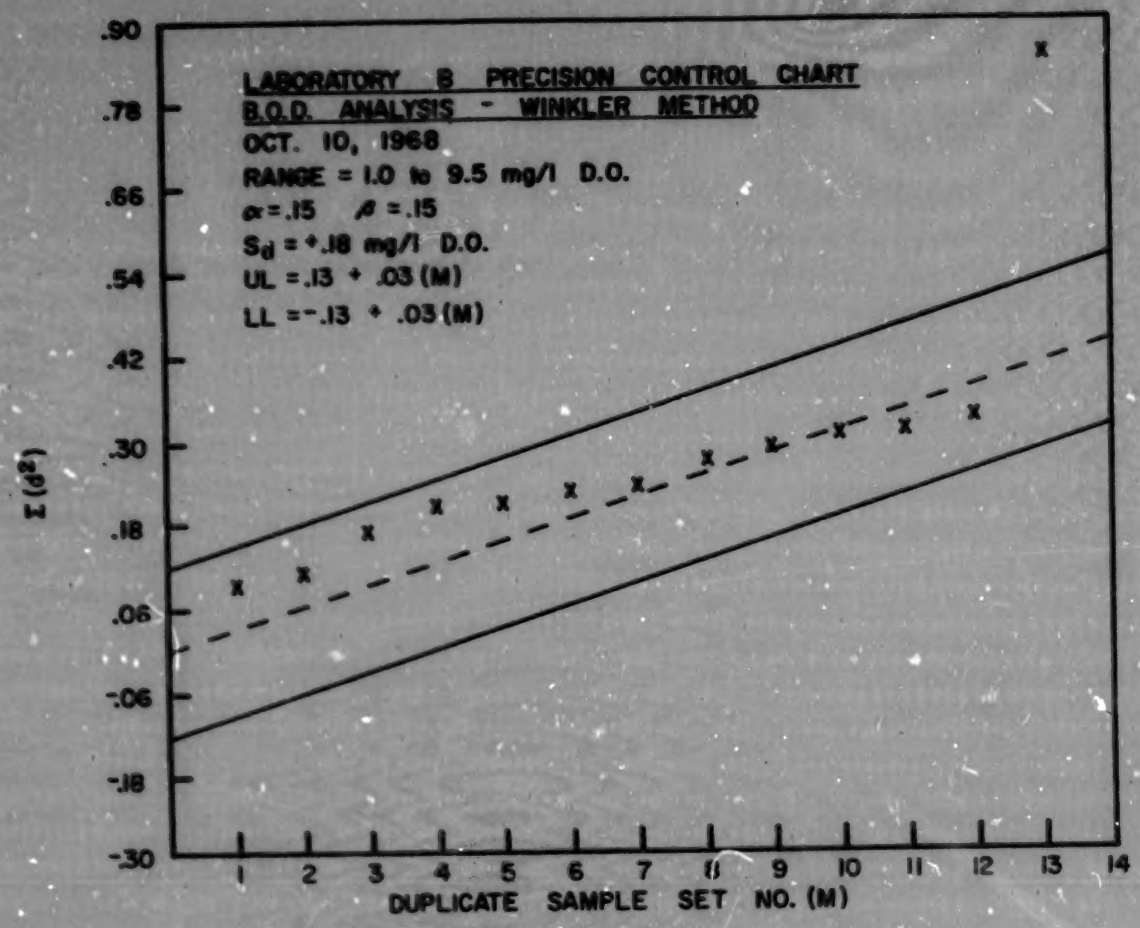


ANALYSIS OUT OF CONTROL
UPPER LIMIT

CONTINUOUS ERROR TREND
PROCEDURES:
SAME AS ABOVE BUT STOP
ANALYSIS WHEN TREND IS
DETECTED.

LABORATORY QUALITY CONTROL CHARTS

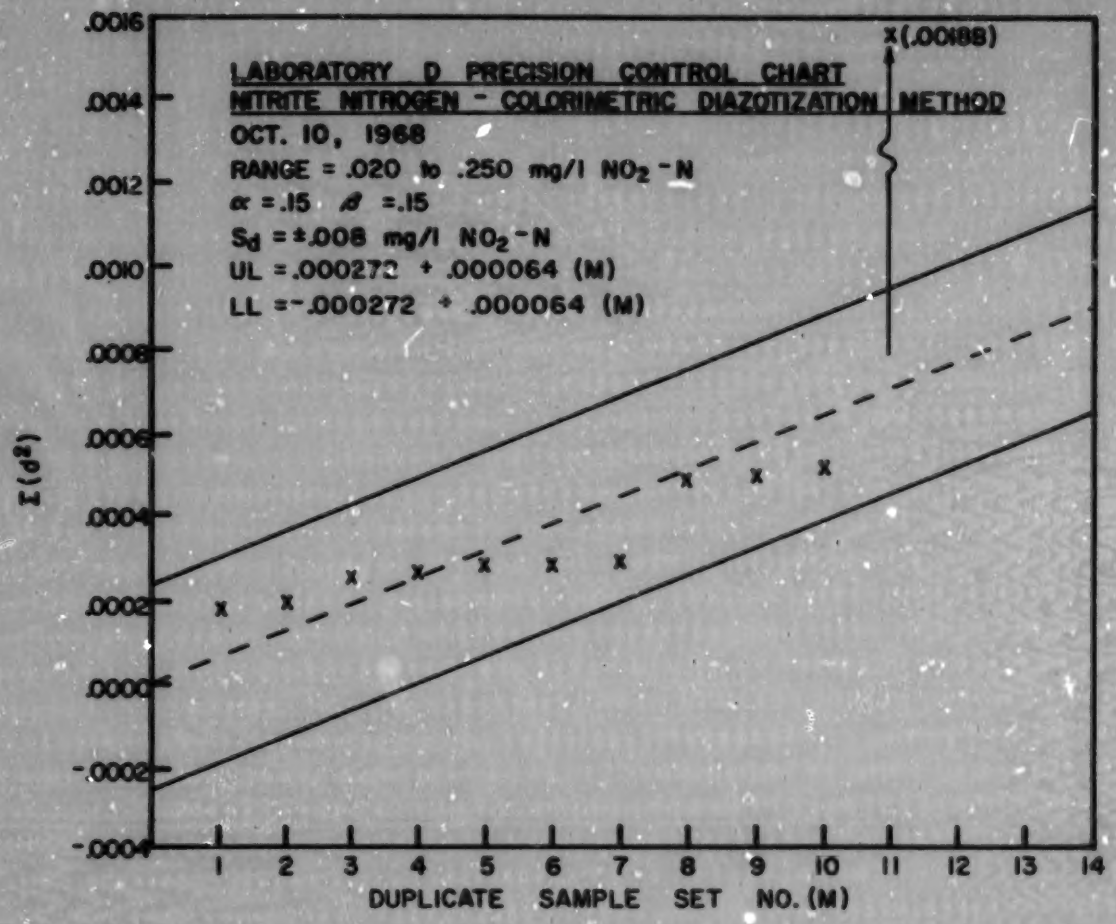
ILLUSTRATIONS OF CONTROL CHARTS



CORRECTIVE PROCEDURES:

1. STOP ANALYSIS AT SAMPLE SET 13
2. LOCATE CAUSE OR ASSUME CHANCE CAUSES
3. CORRECT PROBLEM IF POSSIBLE
4. SAMPLES CANNOT BE RERUN ON B.O.D. - REJECT ALL DATA BETWEEN SAMPLE SETS 12 & 13
5. IF DUPLICATES ARE RUN ON ALL SAMPLES - REJECT SAMPLE SET 13
6. BEGIN PLOTTING $\Sigma(d^2)$ AT SAMPLE SET 1

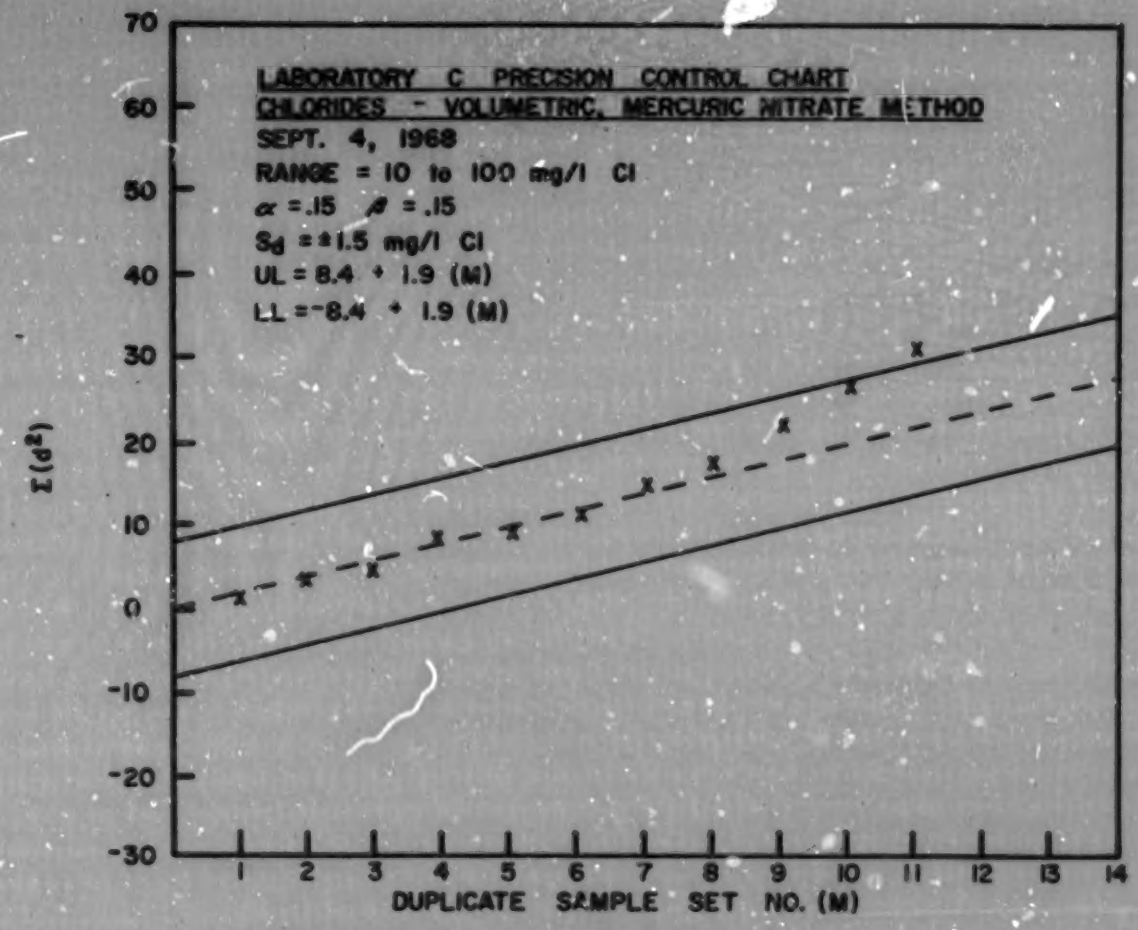
OUT OF CONTROL ON UPPER LIMIT



CORRECTIVE PROCEDURES:

1. STOP ANALYSIS AT SAMPLE SET 11
2. LOCATE CAUSE OR ASSUME CHANCE CAUSES
3. CORRECT PROBLEM
4. RERUN SAMPLES BETWEEN SAMPLE SETS 10 AND 11
5. BEGIN PLOTTING $\Sigma(d^2)$ AT SAMPLE SET 1

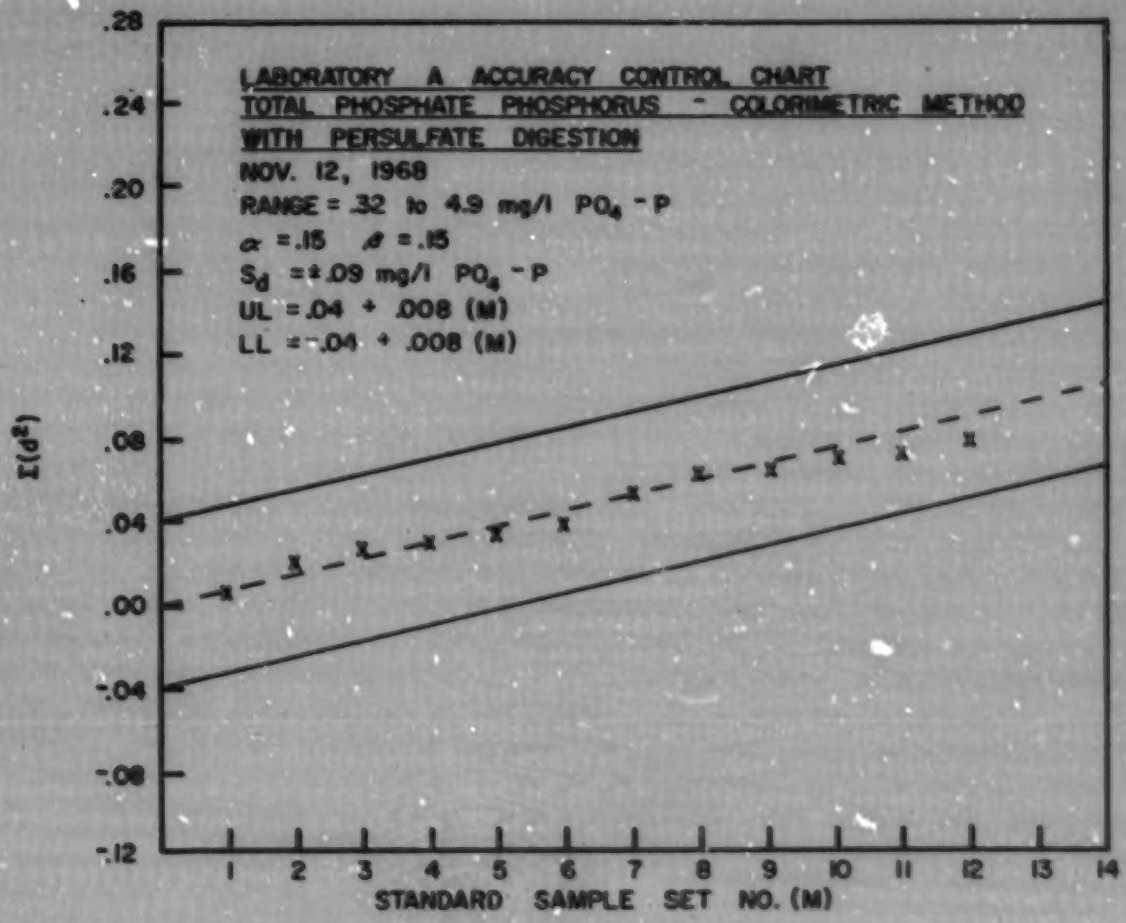
OUT OF CONTROL ON UPPER LIMIT



CORRECTIVE PROCEDURES:

1. ANALYSIS COULD HAVE BEEN STOPPED AT SAMPLE SET 9 OR 10
2. ANALYSIS DEFINITLY STOPPED AT SAMPLE SET 11
3. LOCATE CAUSE
4. CORRECT PROBLEM
5. IF ANALYSIS STOPPED AT SAMPLE SET 9 - RERUN SAMPLES BETWEEN SAMPLE SETS 8 & 9
6. IF ANALYSIS STOPPED AT SAMPLE SET 10 - RERUN SAMPLES BETWEEN SAMPLE SETS 9 & 10
7. IF ANALYSIS STOPPED AT SAMPLE SET 11 - RERUN SAMPLES BETWEEN SAMPLE SETS 10 & 11
8. IN ANY CASE BEGIN PLOTTING $\Sigma(d^2)$ AT SAMPLE SET 1

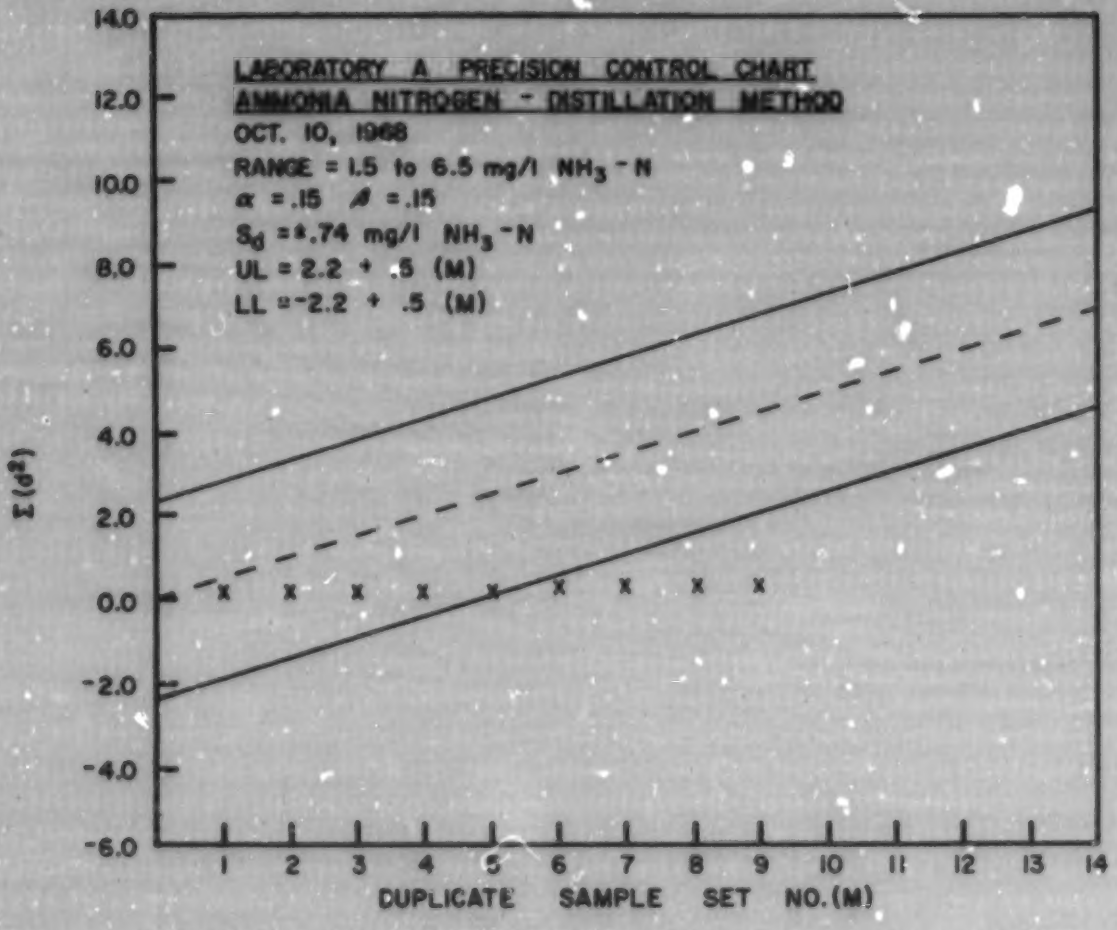
OUT OF CONTROL ON UPPER LIMIT
(CONTINUOUS ERROR TREND)



CORRECTIVE PROCEDURES:

1. NO PROBLEMS
2. CONTINUE ANALYSIS

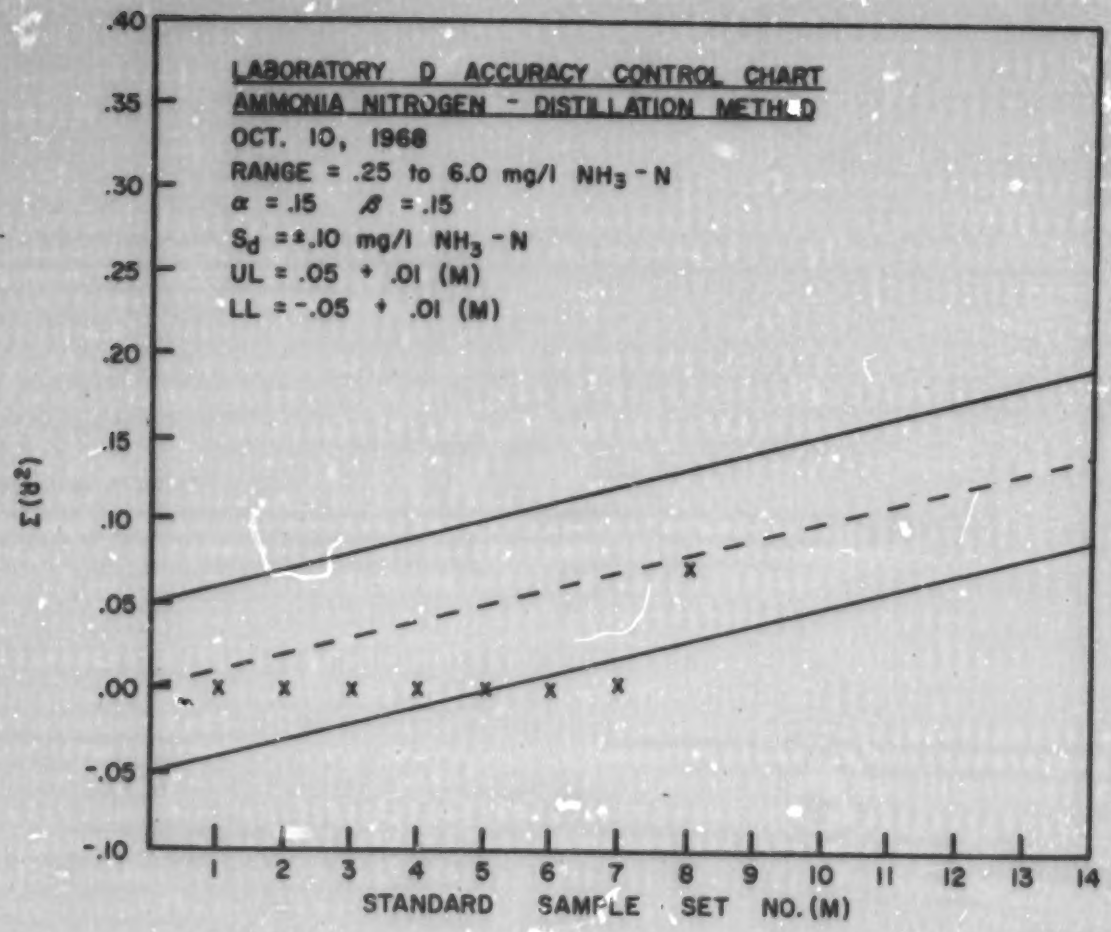
IN CONTROL



CORRECTIVE PROCEDURES:

1. CONTINUE ANALYSIS BEYOND SAMPLE SET 6 THROUGH SERIES OF SAMPLES UNLESS TREND CHANGES
2. ASSUME INCREASED EFFICIENCY
3. CONSTRUCT NEW CHART ON RECENT DATA
4. BEGIN PLOTTING $\Sigma(d^2)$ AT SAMPLE SET 1
5. OBSERVE ANALYST FOR FALSE REPORTING

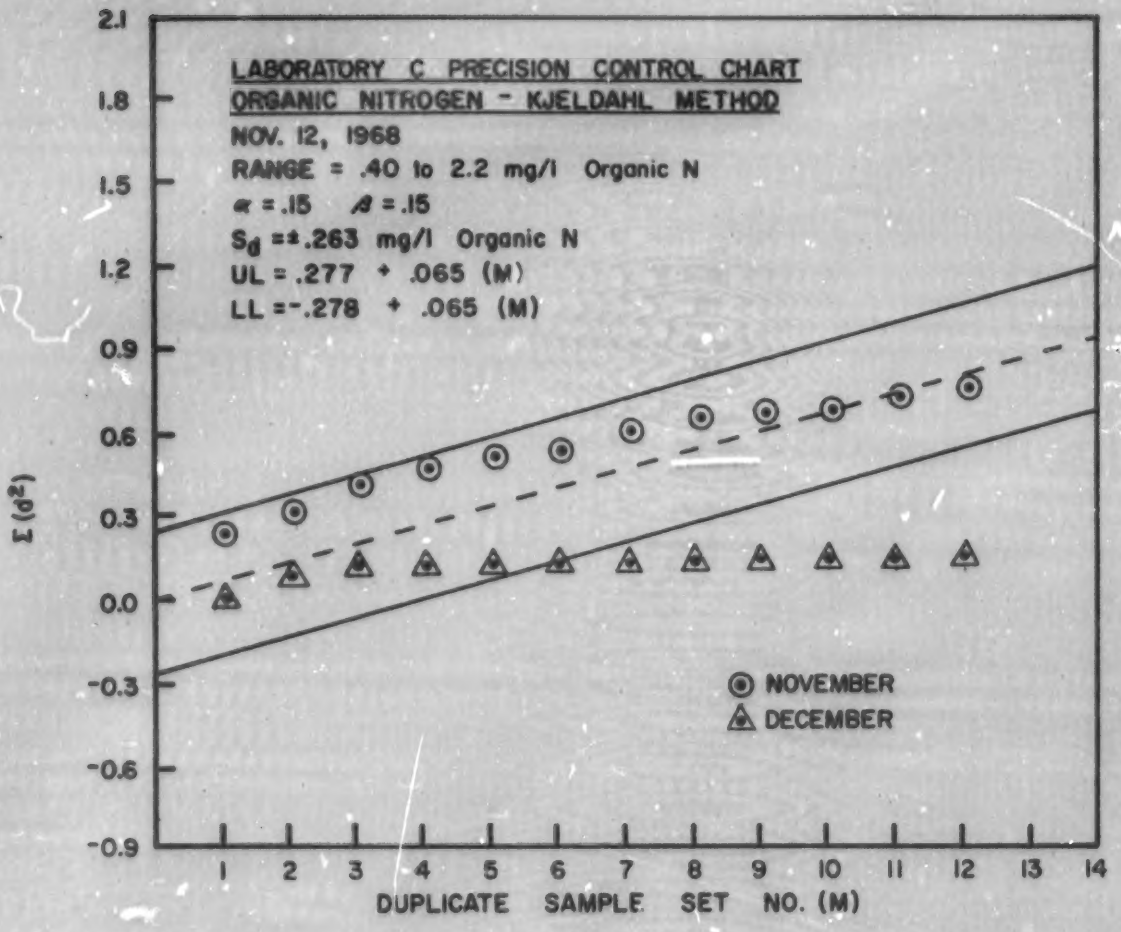
OUT OF CONTROL ON LOWER LIMIT
(CONTINUOUS TREND)



CORRECTIVE PROCEDURES:

1. STOP CHARTING AT SAMPLE SET 8
2. BEGIN NEW CHART BY PLOTTING $\Sigma(d^2)$ OF SAMPLE SET 8 AT SAMPLE SET 1
3. OBSERVE OPERATIONS FOR POSSIBLE PROBLEMS
4. CONTINUE ANALYSIS WITH CAUTION

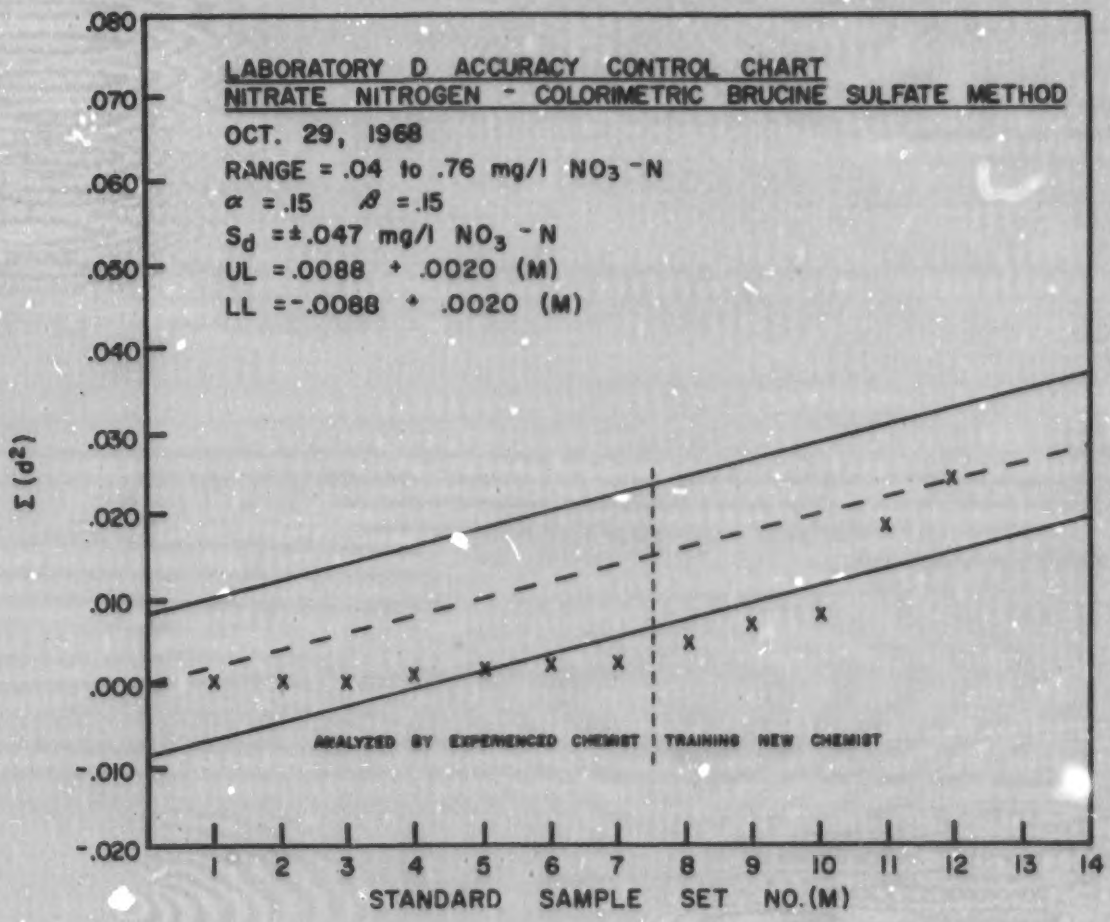
OUT OF CONTROL ON LOWER LIMIT
(CHANGE OF TREND)



CORRECTIVE PROCEDURES:

1. NOVEMBER DATA IN CONTROL
2. DECEMBER DATA PLOTTED ON SAME CHART
3. CONTINUE ANALYSIS BEYOND SAMPLE SET 6 THROUGH DECEMBER SAMPLES
4. CONSTRUCT NEW CHART ON RECENT DATA
5. PLOT $\Sigma(d^2)$ ON NEW CHART FOR JANUARY SAMPLES

OUT OF CONTROL ON LOWER LIMIT
(INCREASED EFFICIENCY)



CORRECTIVE PROCEDURES:

1. SAMPLES 1 THROUGH 7 ANALYZED BY EXPERIENCED CHEMIST
2. TRAINING OF INEXPERIENCED CHEMIST BEGAN AT SAMPLE SET 8
3. TRAINING CONTINUED THROUGH SAMPLE SET 11
4. INEXPERIENCED CHEMIST TOOK COMPLETE CONTROL ON SAMPLE SET 12

EFFECT OF TRAINING ON CONTROL CHARTS

OPERATING

CONTROL CHARTS

DATA CARD AND MASTER LOG SYSTEM

An analytical laboratory must have an orderly and efficient system of handling data. This insures the legal defensibility and validity of the data produced in the laboratory. The FWPCA has successfully used such a system over the past several years. Referred to as the data card and master log system, it is composed of two parts:

1. The data cards for recording all raw data and computations made by the analyst
2. The master log for recording a summary of validated data.

The data cards have a consecutive serial number for each parameter being analyzed. All cards are issued by the laboratory supervisor and are accountable. The entire operation of arriving at a value through the various methods of analyses and mathematical calculations is recorded directly on the data cards, step-by-step. The analyst is not to recopy raw data from any other source onto the cards. To insure permanency of these raw data, permanent ink should be used on the data cards. Completed data cards are to be returned to the laboratory supervisor for data validation.

The master log is a bound book with pages arranged in original and tear-out copy order. Page sets are numbered consecutively. The laboratory supervisor records the validated data in the master log

book. Upon completion of a page in the data book, the tear-out copy page is removed and used as a working data sheet by the project director.

Upon completion of a project the numbered data cards and master log book are stored together for safe keeping and future referral.

ILLUSTRATIONS

of

DATA CARDS

SAMPLE SOURCE (1) DETERMINATION (4)

METHOD (5)

ANALYST (2) REFERENCE (6)

DATE COLLECTED (3)

DATE	SAMPLE NUMBER	ALIQUOT	O.D. or %TRANS.		Mg./ALIQUOT		FACTOR	Mg/l	µg/l
(7)	(8)	(9)	(10)		(11)	(12)	(13)	(14)	(15)

KRC-27 RECORD OF COLORIMETRIC DATA

Key to Annotated Items:

Place in this space

(1) The name of the project or the precise location where sample was collected

(2) The signature of the person analyzing the sample

(3) The date the sample was collected

(4) The parameter being analyzed

(5) The name of the analytical method being used to analyze the sample

(6) The name of the publication that lists the method being used in the analysis of the sample, such as *Standard Methods*, 12th edition, 1965

(7) The date the analysis was performed

(8) The number assigned to the individual sample

(9) The number ml. used in the analysis

(10) The optical density or the % transmittance of the sample on a spectrophotometer

(11) The value of the reading from item (10) taken from a standard curve

(12) Blank space to be used at the analyst's discretion

(13) A number arrived at by dividing the number of ml. of sample used in the analysis, into the total number of ml. of liquid required for the analysis, "the dilution factor"

(14) The value obtained by multiplying item (11) times item (13)

(15) The final value in micrograms/liter if desired

SAMPLE SOURCE Big CHEMICAL CO. OUTFALL
ANALYST Jim Bear
DATE COLLECTED 3-10-68

DETERMINATION NO_3-N (Nitrate)
METHOD MODIFIED BRUCINE
REFERENCE EWPCA OFFICIAL METHOD

DATE	SAMPLE NUMBER	ALIQUOT	O.D. or %TRANS.		Mg./ALIQUOT		FACTOR	Mg/1	µg/1
3-10-68	S-1	10ml	.200		2.31		1	2.3	
"	S-2	1ml	.215		2.40		10	24.0	
"	S-3	5ml	.100		1.30		2	2.6	

KRC-27 RECORD OF COLORIMETRIC DATA

SAMPLE SOURCE (1)

ANALYST (2)

DATE COLLECTED (3)

DATE (4)	SAMPLE NUMBER (5)	(6)							

RECORD OF MISC. SAMPLE DATA

KRC-25
October, 1968

Key to Annotated Items Above:

Place in this space

(1) The name of the project or the particular location where sample was collected

(2) The signature of the person analyzing the sample

(3) The date the sample was collected

(4) The date the sample was analyzed

(5) The number assigned to the individual sample

(6) The parameter being analyzed

SAMPLE SOURCE
SALT CREEK PROJECT
ANALYST [illegible]
DATE COLLECTED 5-10-68

0083

DATE	SAMPLE NUMBER	pH	SPECIFIC CONDUCTANCE						
5-10-68	S-1	7.2	7,400						
"	S-2	7.3	7,000						
"	S-3	4.5	10,000						

KRC-25
October, 1968

RECORD OF MISC. SAMPLE DATA

Sample Source (1) _____ Determination (4) _____

_____ Method (5) _____

Analyst (2) _____ Reference (6) _____

Calculation (3) _____ Date Collected (7) _____

Date	Sample Number	Aliquot	Flask Number	Titer	Blank Correction	Corrected Titer	Factor	Mg./L	
(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	

DETERMINATION OF VOLUMETRIC TITRATION

KRC-29
Sept. 1967

Key to Annotated Items Above:

Place in this space

(1) The name of the project or the particular location where sample was collected

(2) The signature of the person analyzing the sample

(3) A brief formula of the method used to obtain the final mg/l in item 16

(4) The parameter being analyzed

(5) The name of the analytical method used to analyze the sample

(6) The name of the publication that lists the analytical method being used

(7) The date the sample was collected

(8) The date the sample was analyzed

(9) The number assigned to the individual sample

(10) The number of ml. used in the analysis

(11) The number of the flask or etc. used to titrate the sample

(12) The number of ml. of titrant used in titrating the sample

(13) The number of ml. used to obtain an end point of a blank

(14) The value obtained by subtracting item (13) from item (12)

(15) A number obtained by dividing the number of ml. of sample used in the analysis into the total number of ml. of liquid used in the analysis, "the dilution factor"

(16) The value obtained by multiplying item (14) by item (15)

Sample Source JONES CREEK AT HWY 99
Analyst Joe Smith.
Calculation $Cl^- = (Titer - BLK) \times E \times 5$

Determination CHLORIDE
Method MERCURIC NITRATE
Reference EWPCA OFFICAL METHOD
Date Collected 10-9-68

Date	Sample Number	Aliquot	Flask Number	Titer	Blank Correction	Corrected Titer	Factor	Mg./L
68 10-10	S-1	100ml.	1	5.10	.10	5.00	5	25
	S-2	50ml.	8	9.15	.10	9.05	10	91
	S-3	.5ml.	13	6.10	.10	6.00	1000	6000

DETERMINATION OF VOLUMETRIC TITRATION

KRC-29
Sept. 1967

KRC-26
June 1967

SOLIDS DETERMINATION

Sample Source (1) Date (3)

Analyst (4)

Sample Collected (2)

SAMPLE	(5)			
VOLUME ml	(6)			
DISH NUMBER	(7)			
GROSS WEIGHT gm	(8)			
ASHED WEIGHT gm	(9)			
TARE WEIGHT gm	(10)			
RESIDUE gm	(11)			
VOLATILE RESIDUE gm	(12)			
FACTOR	(13)			
TOTAL S. SOLID Mg/1	(14)			
T. SUSPENDED VOLATILE SOLIDS Mg/1	(15)			
TOTAL SOLID Mg/1	(16)			
T. VOLATILE SOLIDS Mg/1	(17)			
TOTAL D. SOLID Mg/1	(18)			

Key to Annotated Items on the Solids Card:

Place in this space

(1) The name of the project or the particular location where sample was collected

(2) The date the sample was collected

(3) The date the sample was analyzed

(4) The signature of person analyzing the sample

(5) The number assigned to the individual sample

(6) The number of ml. used in the analysis

(7) The number of the container used for the analysis

(8) The weight of the container plus the residue remaining after drying treatment

(9) The weight of the container plus the residue remaining after the 600°C heat treatment

(10) The original dry weight of the container

(11) The value obtained by subtracting item (10) from item (8)

(12) The value obtained by subtracting item (9) from item (8)

(13) The value obtained by the formula $\frac{1000}{\text{Item (6)}} \times 1000$

(14) The value obtained by multiplying item (13) times item (11) if total suspended solids are analyzed

(15) The value obtained by multiplying item (13) times item (12) if total suspended volatile solids are analyzed

(16) The value obtained by multiplying item (13) times item (11) if total solids are analyzed

(17) The value obtained by multiplying item (13) times item (12) if total volatile solids are analyzed

(18) The value obtained by subtracting item (14) from item (16)

KRC-26
June 1967

08000

SOLIDS DETERMINATION

Sample Source BLUE RIVER Project Date 3-6-59
Sample Collected 3-5-59 Analyst Sam Sakai

SAMPLE	S-1	S-2	S-3	
VOLUME ml	10	25	100	
DISH NUMBER	8	17	3	
GROSS WEIGHT gm	23.4010	23.9210	22.1783	
ASHED WEIGHT gm	23.3010	23.9210	22.1000	
TARE WEIGHT gm	23.3000	23.5110	22.1000	
RESIDUE gm	.1010	.4100	.0783	
VOLATILE RESIDUE gm	.1000	.0000	.0753	
FACTOR	100,000	40,000	10,000	
TOTAL S. SOLID Mg/l				
T. SUSPENDED VOLATILE SOLIDS Mg/l				
TOTAL SOLID Mg/l	10,100	16,400	783	
T. VOLATILE SOLIDS Mg/l	10,000	0	783	
TOTAL D. SOLID Mg/l				

Rev.
KRC-28
Mar. 1968 BIOCHEMICAL OXYGEN DEMAND AT 20°C

SAMPLE SOURCE (1) _____ DATE (3) _____
_____ ANALYST (4) _____
DATE COLLECTED (2) _____

SAMPLE	(5)						
% CONCENTRATION	(6)						
DAYS INCUBATED	(7)						
TIME	(8)						
BOTTLE #	(9)						
DISSOLVED OXYGEN INITIAL	(10)						
BOTTLE #	(11)						
DISSOLVED OXYGEN FINAL	(12)						
	(13)						
ACTUAL DEPLETION	(14)						
BLANK CORRECTION	(15)						
CORRECTED DEPLETION	(16)						
DILUTION FACTOR	(17)						
B.O.D. mg/l	(18)						
B.O.D. mg/l	(19)						

Key to Annotated Items on Biochemical Oxygen Demand Card:

Place in this space

(1) The name of the project or the particular location the sample was collected

(2) The date the sample was collected

(3) The date the BOD was set up

(4) The signature of the person analyzing the sample

(5) The number assigned to the individual sample

(6) The % dilution of the sample

(7) The number of days the sample incubated

(8) The time the BOD was set up

(9) The bottle number of the initial DO

(10) The value of the initial DO in mg/l

(11) The bottle numbers of the two samples to be incubated

(12) The final DO value of the two incubated bottles

(13) The average of the values in item (12)

(14) The value obtained by subtracting the value of item (13) from item (10)

(15) The seed correction value. Used only when the sample was seeded

(16) The value obtained by subtracting item (15) from item (14)

(17) The value obtained by dividing the % sample used into 100%

(18) The value obtained by multiplying item (17) by item (18)

(19) The BOD value to be reported

00.000

KRC-28
July 12, 1968 BIOCHEMICAL OXYGEN DEMAND AT 20°C

Sample Source JUMPER CREEK Date 5-10-68
Project Analyst Jas. Ener
Date Collected 5-10-68

SAMPLE	S-1	S-1	S-1	S-2	S-2	S-2
% CONCENTRATION	100	50	25	50	25	10
DAYS INCUBATED	5	5	5	5	5	5
TIME	2:00PM →					
BOTTLE NUMBER	341	376	348	410	315	118
DISSOLVED OXYGEN INITIAL	8.15	8.10	8.20	7.95	7.90	8.70
BOTTLE NUMBER	350 / 311	301 / 302	111 / 52	13 / 195	35 / 1	300 / 358
DISSOLVED OXYGEN FINAL	1.03 / 1.01	4.55 / 4.65	6.45 / 6.45	.50 / .61	2.59 / 2.61	6.45 / 6.55
	1.02	4.60	6.45	—	2.60	6.50
ACTUAL DEPLETION	7.13	3.50	1.75	—	5.30	2.20
BLANK CORRECTION	—	—	—	—	—	—
CORRECTED DEPLETION	7.13	3.50	1.75	—	5.30	2.20
DILUTION FACTOR	1	2	4		4	10
B.O.D. Mg/L	7.13	7.00	7.00		21.2	22.0
B.O.D. Mg/L	7				21	

MASTER LOG

BOOK

TECHNICAL SERVICES PROGRAM

ROBERT S. KERR WATER RESEARCH CENTER

CHEMICAL DATA

PROJECT

BLUE RIVER WATER QUALITY STUDY

Date:

STARTED JAN. 5, 1969

COMPLETED Feb. 10, 1969

NOTES

1. THE COD SAMPLES WERE PRESERVED WITH 1ml. CONC. H_2SO_4. ALL OTHERS WERE ICED UPON COLLECTION.

2. SAMPLE NO. 1-4 WERE TAKEN AT 8:00 AM ±30MIN.
   SAMPLE NO. 5&6 WERE TAKEN AT 11:30 AM ±15 MIN.
   SAMPLE NO. 7&8 WERE TAKEN AT 3:00 PM ± 15MIN.
   SAMPLE NO. 9&10 WERE TAKEN AT 6:30PM ± 15MIN.

PROJECT: BLUE RIVER
PROJECT LEADER: JOE JONES

CHEMISTRY DATA

Date Collected: JAN. 1-4-9-20, 1969 Validated by: Jim Smith

Date Analyzed	Sample Number	BOD	COD	$SO_4^=$	Cl^-	TDS				
69										
1-2	BR-1	10	18	113	250	310				
	BR-2	5	10	112	240	311				
	BR-3	4	9	113	245	320				
	BR-4	6	11	104	249	315				
1-4	BR-5	9	18	110	253	312				
	BR-6	7	16	112	241	316				
1-10	BR-7	9	16	107	235	312				
	BR-8	7	12	115	250	310				
1-20	BR-9	10	20	116	230	315				
	BR-10	15	25	112	232	307				

All chemical analyses expressed as mg/l, except pH and unless otherwise indicated.

PROJECT: BLUE RIVER
PROJECT LEADER: JOE JONES

COPY

CHEMISTRY DATA

Date Collected: JAN. 1-4-9-20, 1969 Validated by: Jim Smith

Date Analyzed	Remote Number	BOD	COD	$SO_4^=$	Cl^-	TDS				
69 1-2	BR-1	10	18	113	250	310				
	BR-2	5	10	112	240	311				
	BR-3	4	9	113	245	320				
	BR-4	6	11	104	249	315				
1-4	BR-5	9	18	110	253	312				
	BR-6	7	16	112	241	316				
1-10	BR-7	9	16	107	235	312				
	BR-8	7	12	115	250	310				
1-20	BR-9	10	20	116	230	315				
	BR-10	15	25	112	232	307				

All chemical analyses expressed as mg/l, except pH and unless otherwise indicated.

AN ANALYTICAL QUALITY CONTROL PROGRAM FOR EFFICIENT LABORATORY MANAGEMENT*

by

R. E. Crowe, R. Harkins, J. Kingery, and B. G. Benefield**

Introduction

Quality control procedures in general have been used since man began his thinking process. Galileo, in his experiments to determine the surface tensions of liquids, gave detailed instructions for obtaining a consistent set of results (1). The artisan guilds of the Middle Ages prescribed extended apprenticeships before a person was considered a master craftsman. This training maintained a level of competence within the guild (2). Dr. A. Shewhart of Bell Telephone Laboratories developed the basic theory of control charts in the 1920s (3). This was the beginning of industrial use and acceptance of these and other statistical techniques to measure the quality of products of a manufacturing process. The development of these techniques in industry led to their limited use in the analytical laboratory (4).

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* A paper scheduled for presentation at the 20th Annual Oklahoma Industrial Waste and Pollution Control Conference, Oklahoma State University, Stillwater, March 31-April 1, 1969.

** Respectively, Chief, Chemistry and Biology Section, Technical Assistance, Technical Services Program; Acting Chief, Pollution Surveillance, Technical Services Program; Mathematical Statistician, Pollution Surveillance, Technical Services Program; and Chemist, Chemistry and Biology Section, Technical Assistance, Technical Services Program, all of the Robert S. Kerr Water Research Center, Federal Water Pollution Control Administration, U. S. Department of the Interior, Ada, Oklahoma.

Although laboratory operations are not considered to be manufacturing processes, it can be recognized that the analytical data produced by any laboratory are, in actuality, the products of that process. As with industry, a quality control program should be employed in the laboratory to insure the quality of its products, which in turn, characterize the normal laboratory operations, and detect abnormal operations when they occur. This paper presents one such program for consideration as a tool in characterizing a laboratory's operations and maintaining quality control in the laboratory.

Laboratory "Fingerprint"

Anyone who has worked in, supervised, or managed the operations of an analytical laboratory is well aware of the basic tools for determining quality of data produced. These tools are duplicate sample analyses and spiked or standard sample analyses. These have been used to indicate the precision and accuracy of the process producing the data.

Those who have used these tools recognize the difficulty involved in making a decision as to the validity of the data produced based on duplicate and spiked or standard sample analyses. The common practice has been to visually observe the data and arbitrarily judge their acceptability with no concrete basis for the decision. Obviously, it would be advantageous to have a so-called "fingerprint" of the precision and accuracy for the normal operations of a specific laboratory group for the analysis of a specific parameter.

"Fingerprints" of this nature can tell us many things about the laboratory's operations. For example, they could tell us when problems exist with the analysts, reagents, glassware, instruments, etc. They could indicate whether the laboratory is operating normally or abnormally, thus pointing out when data generated should be accepted, questioned, or rejected. In addition, they could tell us when the laboratory is operating at optimum efficiency. As with industry, the laboratory sometimes generates products which are not acceptable. In these cases, samples must be analyzed again to produce acceptable results. These "fingerprints" could help us determine which samples or sets of samples should be reanalyzed.

The laboratory "fingerprints" we refer to are in the form of control charts. The construction and use of such charts are discussed below.

Construction of Control Charts

As we have indicated, two control charts are required to "fingerprint" the laboratory operations for a given analytical procedure. These are referred to as precision and accuracy control charts. Precision control charts are constructed from duplicate sample analyses data; accuracy control charts are constructed from spiked or standard sample analyses data. A set of the two represents, and is restricted to, a specific laboratory, group of analysts, analytical method, range of concentration and period of time. To construct the precision and accuracy control charts, it is necessary to obtain several sets of duplicate and spiked

or standard sample data. The greater the number of sets of initial data obtained, the better the "fingerprint" of the laboratory operations. Economics must be considered, however, in obtaining the initial sets of data. It is recommended that at least 20 sets of duplicate and 20 sets of spiked sample data from an in-control process be used to initially construct the control charts. The selection of in-control data can be a judgment decision or, if desired, extreme values can be systematically eliminated by the Dixon and Massey method (5) of processing data for extreme values.

The initial sets of data must be obtained under the following conditions:

1. Normal laboratory operations
2. Constant analyst or group of analysts
3. Consistent method
4. Narrow range of concentration of the parameter analyzed.

The reasons for the first three conditions are obvious; number 4 needs more explanation.

The precision and accuracy of the analyses for many parameters are proportional to the concentration of the parameter to be measured. This may require the use of several control charts in varying ranges of concentrations for a given parameter. Only experience will dictate this. It is important to note here that there is complete control over the range of concentration of spiked samples or standard samples, but little

or no control over the range of concentration of the duplicate samples.

It is important also to point out the basic differences between a spiked and a standard sample. These terms have been used synonymously at times in the discussion of accuracy data; however, the two differ greatly. A spiked sample can be defined as an environmental sample to which has been added or "spiked" a known quantity of that parameter already present in the sample in significant concentrations. The environmental sample obviously must be analyzed before as well as after the "spiking" of the sample. Spiked samples should be used in situations where knowledge is insufficient as to the interferences of the method or of the environment from which the sample was obtained. This situation might also necessitate a complete and independent study of the interferences.

A standard sample can be defined as one prepared by adding a known concentration of a given parameter to distilled water. The sample should then be analyzed identically to the environmental samples. A standard sample can be used in situations where the interferences of the method with the environment are sufficiently known. In other words, a standard sample can be used where interferences of the method are not questioned, and the assumption made that interferences are absent. A standard sample has the inherent disadvantage of unusual appearance, so that the analyst is aware of its introduction into a series of environmental samples. This could create bias in the results.

The control charts are derived from three basic calculations. No attempt is made here to develop the mathematics upon which the control charts are based. However, references are given so that those who wish to delve more deeply into the subject may do so (6).

These basic calculations are:

1. Standard deviation of the differences between duplicates, or in the case of spiked or standard samples, between the known quantity and the quantity obtained.
2. The upper control limit
3. The lower control limit.

Prior to these calculations, two decisions must be made:

1. The α and β levels
2. The allowable variability levels

By definition, α is the probability of judging the process to be out-of-control, when in fact, it is in-control. It is recommended that α be chosen to lie within the boundaries of .05 and .15; that is, the laboratory personnel are willing to stop the laboratory process somewhere between 5 and 15 percent of the time, judging it to be out-of-control, when in fact, it is in-control. If the cost of examining a process to determine the reason or reasons for being out-of-control is considerable, then it may be desirable to choose a low α. Likewise, if the cost is negligible, it may be desirable to choose a larger α value and thus stop the process more frequently.

On the other hand, β is defined as the probability of judging the process to be in-control when it is not. Again, it is recommended that β be chosen to lie between the values of .05 and .15; thus, the laboratory personnel are willing to accept out-of-control data somewhere between 5 and 15 percent of the time. The economic considerations used in choosing α also apply in choosing β. The effects of varying α and β are demonstrated in Figure 1.

It is also essential to set maximum and minimum allowable variability levels. It is necessary to specify a value for the minimum and maximum amount of variation that will be allowable in the system. These minimum and maximum amounts are referred to as σ_o^2 and σ_1^2 respectively. The values used should be based on a knowledge of the variation in the procedure under consideration. However, if no such knowledge is available, the values may be arbitrarily set at $\sigma_o^2 = (\sigma - .20\sigma)^2$ and $\sigma_1^2 = (\sigma + .20\sigma)^2$.

$$S_d^2 = \frac{\sum_{i=1}^{n} di^2 - \frac{(\sum_{i}^{n} di)^2}{N}}{N - 1} = \text{Variance of the differences}$$

$$S_d = \sqrt{S_d^2} = \text{Standard deviation of the differences} \qquad (1)$$

$S_o^2 = (.8S_d)^2$ estimates σ_o^2

$S_1^2 = (1.2S_d)^2$ estimates σ_1^2

$$UL(M) = \frac{2 \log_e\left[\frac{1-\beta}{\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} \qquad (2)$$

$$LL(M) = \frac{2 \log_e\left[\frac{\beta}{1-\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} \qquad (3)$$

Where: UL(M) = upper limit at M sets of duplicate or spiked samples

LL(M) = lower limit at M sets of duplicate or spiked samples

di = the difference between the i^{th} set of duplicates or spiked samples

N = the total number of sets of duplicates or spiked samples used to construct the control charts

S_o^2 = minimum amount of variation allowed in the system

S_1^2 = maximum amount of variation allowed in the system

α = percent of time you are willing to judge the procedure out-of-control when it is in-control

β = percent of time you are willing to judge the procedure in-control when it is out-of-control

M = number of sets of duplicates or spiked samples used in calculating the value to be plotted on the chart

For clarification purposes, an example of using the above equations in making the calculations is given below. The example involves the measurement of total phosphate phosphorus by the colorimetric, with persulfate digestion, method. Twenty-three sets of standards at concentrations

varying from .32 to 4.9 mg/l of total phosphate phosphorus were used in the calculations. It was assumed that there was no appreciable proportional error in this range of concentration. Also, by visual observation we did not reject any data as being out of control.

Results of Analyses of Standards

(mg/l Total PO_4-P)

Actual	Obtained	Difference (di)	di^2
.34	.33	+.01	.0001
.49	.49	.00	.0000
.49	.49	.00	.0000
.68	.65	+.03	.0009
.67	.65	+.02	.0004
.66	.70	-.04	.0016
.83	.80	+.03	.0009
.34	.34	.00	.0000
.50	.47	+.03	.0009
.40	.40	.00	.0000
.50	.53	-.03	.0009
.66	.60	+.06	.0036
.50	.56	-.06	.0036
.52	.59	-.07	.0049
.98	.75	+.23	.0529
.49	.63	-.14	.0196
1.6	1.7	-.10	.0100
1.3	1.2	+.10	.0100
3.3	3.3	.00	.0000
4.9	4.6	+.30	.0900
2.3	2.3	.00	.0000
1.3	1.3	.00	.0000
2.3	2.4	-.10	.0100

$$\Sigma di = .27$$
$$\Sigma di^2 = .21$$
$$(\Sigma di)^2 = .07$$

$$S_d^2 = \frac{\Sigma di^2 - \frac{(\Sigma di)^2}{N}}{N-1} = \frac{.21 - \frac{.07}{23}}{22} = .009$$

$$S_d = \sqrt{S_d^2} = \sqrt{.009} = \pm .09 \qquad (1)$$

$$S_o^2 = (.8S_d)^2 = .64S_d^2 = .64(.009) = .006$$

$$S_1^2 = (1.28_d)^2 = 1.44S_d^2 = 1.44(.009) = .013$$

$$UL(M) = \frac{2 \log_e \left[\frac{1-\beta}{\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e \left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$= \frac{3.5}{\frac{1}{.006} - \frac{1}{.013}} + M \frac{\log_e \left[\frac{.013}{.006}\right]}{\frac{1}{.006} - \frac{1}{.013}}$$

$$= \frac{3.5}{90} + M \frac{.69}{90}$$

$$= .039 + .0077(M) \qquad (2)$$

$$LL(M) = \frac{2 \log_e \left[\frac{\beta}{1-\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M \frac{\log_e \left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$= \frac{-3.5}{90} + M \frac{.69}{90}$$

$$= -.039 + .0077(M) \qquad (3)$$

We are now prepared to construct an accuracy control chart. The upper limits on the Y-axis can be calculated using equation (2):

at M = 0

UL (0) = .04 + 0(.008) = .04;

at M = 14

UL (14) = .04 + 14(.008) = .15

These two points can now be plotted to form the upper limit line as shown in Figure 2.

The lower limits on the Y-axis can be calculated using equation (3):

at M = 0

LL (0) = -.04 + 0(.008) = -.04;

at M = 14

LL (14) = -.04 + 14(.008) = .07

These two points can now be plotted to form the lower limit line as shown in Figure 2.

It should be noted that the Y-intercept for the lower control line is the negative of that for the upper control line. This is because α and β are equal. If they are not equal, this condition will not exist.

Figure 2 now represents an accuracy control chart for total phosphate phosphorus which is characteristic of the laboratory operations restricted to the conditions specified on the chart. Only an accuracy control chart has been demonstrated here. The same procedures should be followed to produce a precision control chart.

Use of Control Charts

Now that the control charts have been constructed, we are prepared to plot the values obtained from duplicate and spiked sample results from a series of sample analyses. At this point a decision must be made as to the number of duplicate analyses to be conducted during a series of samples; the same decision must be made on spiked or standard samples. This decision is primarily one of economics.

In considering the number of duplicate and spiked sample analyses to be conducted in a series of samples, it is necessary to weigh the consequences when the data goes out-of-control. The consequences involve reanalyzing a series of samples, or discarding the questionable data obtained. The samples to be reanalyzed should be those lying between the last in-control point and the present out-of-control point. For example, if you have a 100 sample series to be analyzed, and a duplicate and spiked sample are analyzed only once in the series (in the area of the 50th sample) the consequences, if this one sample is out-of-control, are that the first 50 samples must be reanalyzed or discarded.

If all 100 samples were analyzed prior to calculating and plotting the out-of-control samples, then all 100 would need to be reanalyzed or discarded. If duplicate and spiked samples are analyzed more frequently and more realistically, such as at every fifth sample, then it is apparent that, if one sample goes out-of-control, it is necessary to reanalyze only the nine in between the two in-control points, or only five if the laboratory operations are halted at the out-of-control point. In addition, the more frequently the duplicate and spiked samples that are analyzed, the greater the chances of detecting abnormal operations as they occur. Also to be considered are the economics involved in the method used and the stability of the sample. A good example lies in the biochemical oxygen demand (BOD) analysis. It is obvious that this method requires five days, and the sample could not be reanalyzed after that time lapse. For these reasons we conduct duplicate analyses on each BOD analysis and report only those that are in-control.

Once the frequency of duplicate and spiked samples has been determined, it is necessary to prepare spiked or standard samples in concentrations relative to those of the control charts which should be similar to concentrations of the environmental samples. These spiked or standard samples must be intermittently dispersed among the samples of the series to be analyzed and without the analyst's knowledge of concentration. Similarly, duplicate samples must be intermittently dispersed throughout

the series of samples to be analyzed, and ideally without the analyst's knowledge; however, this is sometimes very difficult to accomplish.

It cannot be overemphasized that the results of the duplicate and spiked samples must be calculated immediately upon analyzing the samples. This will allow the $\Sigma(d^2)$ to be plotted as soon as possible on the control charts so that any existing problems can be corrected and samples promptly reanalyzed. A brief and simplified example of these calculations is:

Duplicate Sample No. M	Results No. 1	Results No. 2	Difference (d)	d^2	$\Sigma(d^2)$
1	5.4	5.2	.2	.04	.04
2	4.8	4.7	.1	.01	.05
3	6.1	5.8	.3	.09	.14

Following each calculation, the summation or $\Sigma(d^2)$ is plotted on a chart similar to Figure 2, plotting Σd^2 against the sample number. Upon plotting $\Sigma(d^2)$ one of three possibilities will occur:

1. Out-of-control on the upper limit
2. In-control within the upper and lower limit lines
3. Out-of-control on the lower limit

Each of these possibilities will now be discussed in detail.

There are generally two types of out-of-control on the upper limit conditions. One type is illustrated in Figure 3. This occurs when the laboratory is operating quite normally and suddenly a point goes

out-of-control, usually extremely far from the upper control line. The other type of condition is one in which you have a continuous error trend; in other words, you are approaching an out-of-control condition at a consistent rate, usually forming a trend line in the direction of and at an angle to, the upper control limit line as illustrated in Figure 4. The latter condition (Figure 4) is advantageous over the former since problems can usually be detected early in the continuous error trend condition and corrected before out-of-control actually occurs. The former (Figure 3) case yields no such warning; the operations go out-of-control with no prior indication of a problem. When the operations go out-of-control at the upper limit, obviously the laboratory operations are to be stopped as soon as possible so that the problems can be located and corrected before proceeding with the analysis. Also, keep in mind that it is possible to go out-of-control for no other reason than chance causes. Then the samples in question are to be reanalyzed with duplicate and spiked samples. The first duplicate and spiked sample data are to be plotted, beginning with Sample No. 1 on the control chart. The primary reason for starting at position No. 1 on the control chart is that, since we are plotting the summation of the differences squared, the out-of-control point dominates the next point, thus continuing out-of-control even though it could actually be in-control.

What problems are associated with laboratory operations being

out-of-control? The answer depends upon whether it is a precision control or an accuracy control chart. The causes for out-of-control on the precision chart are usually one or more of the following:

1. The analyst
2. Nature of the sample
3. Glassware contamination

Obviously, we are not concerned over reagents or instrumentation in precision, since analyzing a duplicate sample would duplicate any reagent or instrument error. The analyst is probably the primary source of precision errors; however, the nature of the sample is not to be overlooked. By the nature of the sample we mean the homogeneity of the sample in relation to its amenability to being separated into two equal parts to allow a true duplicate analysis. In the case of samples that contain oils or clumps of insoluble material, it is practically impossible to obtain a duplicate sample. Glassware contamination is probably the least frequent contributor to imprecision; however, it does occur. One flask can be contaminated, where another is not.

Five problems are associated, singly or in combination, with an out-of-control condition on an accuracy control chart. They are:

1. The analyst
2. Glassware contamination
3. Contaminated reagents

4. Instrumentation

5. Sample interference with the spiked material.

All weigh fairly evenly as possible causes of inaccuracy.

The second possibility is where the laboratory is operating in-control within the upper and lower limit lines. This possibility is illustrated in Figure 5. Under these conditions there is no cause for concern over the quality of the data, and samples should be continually analyzed until either a trend develops or a result goes out-of-control at the upper limit.

The remaining possibility is out-of-control on the lower limit as illustrated in Figure 6. This situation is indicative of greater precision and accuracy being attained by a laboratory. This probably would show up in the first plotting on the first control chart, because the more experience the laboratory gains in analyzing a specific parameter by a specific method, the more precise and accurate that laboratory becomes until optimum operation is achieved. When this situation occurs, it is not necessary to stop the analyses; instead, analyzing should continue on the particular sample series involved (unless the trend changes significantly). It is then necessary to construct a new control chart using the latest duplicate and spiked sample data. The new control chart will then represent the current operating characteristics of the laboratory at that time. In the situation of a new laboratory analyzing a new parameter using an unfamiliar method, several charts may be constructed before optimum

operating conditions are attained.

Another reason for an out-of-control on the lower limit occurrence would be the analyst's reporting of false data. This is particularly true with duplicate sample analyses where the analyst is aware of the duplicate sample. This would not be the case with spiked sample analyses since the analyst would not have knowledge of the concentration of the parameter in the standard or spiked sample. If the analyst is suspected of reporting false duplicate data, it would be necessary to mask the duplicate samples so that the analyst is not aware of their presence.

It should also be pointed out that analyzing a duplicate or spiked sample many times with special attention will produce more precise and accurate data than under normal operations. This, in turn, would produce an out-of ontrol on the lower limit condition.

Again, there will be cases where data are out-of-control for no apparent reason. Many such cases can be attributed to chance causes which will occur occasionally.

Standard Deviation

Mentioned previously were the three basic calculations required to "fingerprint" a laboratory's operations. We have discussed the upper and lower control limits. Now let us briefly discuss the third calculation, that of the standard deviation. The purpose of calculating the standard deviation is to allow inter-laboratory comparisons of precision

and accuracy. It also allows similar comparisons with the literature. In this respect, it can be used as a guide to determine if the laboratory is operating "in the ball park" on precision and accuracy for a given parameter. It should be emphasized that the comparisons of standard deviations should be used only as a guide since the standard deviation of a specific laboratory is characteristic of that laboratory's operations and no other.

Summary

Some type of analytical quality control program is necessary for efficient laboratory management and to validate analytical data. Since analytical data are the products of the analytical laboratory, we must know which products to reject and which are to be accepted as valid. The quality of the laboratory products will vary as long as humans are involved in the analyses; therefore, it is important to know when the variations go beyond those occurring under normal laboratory operations so that the end product quality is known.

This paper discusses the construction and use of laboratory "fingerprints", in the form of control charts, to identify and characterize the operations of a laboratory. These "fingerprints" or control charts are limited to the laboratory from which the data are produced. They are also restricted to the conditions under which the samples are analyzed. They were constructed intentionally with the use of basic mathematical equations, thus encouraging their use.

The uses of control charts are detailed. Three possible occurrences when plotting analytical data on the control charts are described, these being:

1. Out-of-control on the upper limit
2. In-control
3. Out-of-control on the lower limit

The charts are interpreted and recommendations made as to what to do when the laboratory operations deviate from normal.

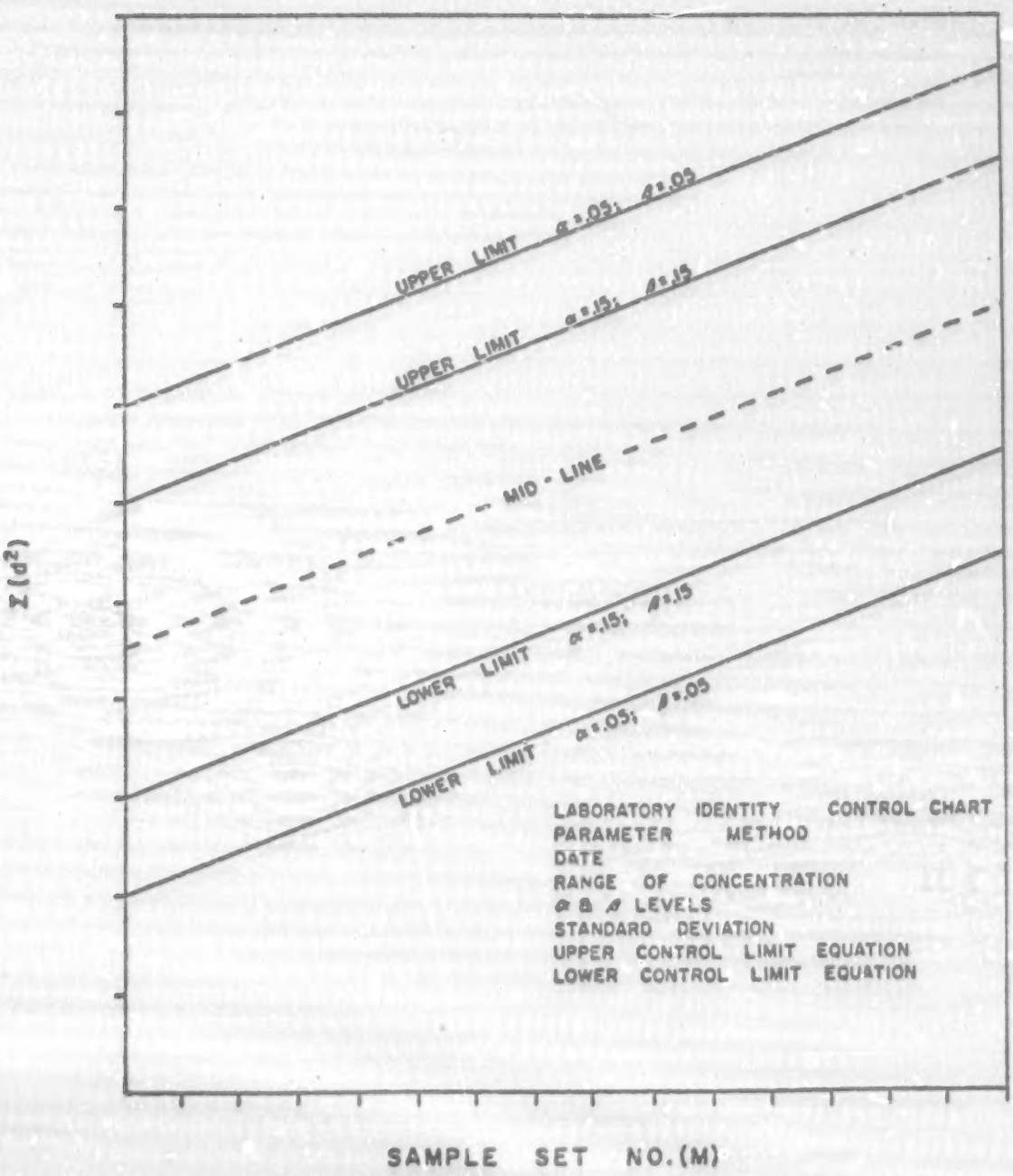

FIGURE 1 - EFFECT OF VARYING α & β

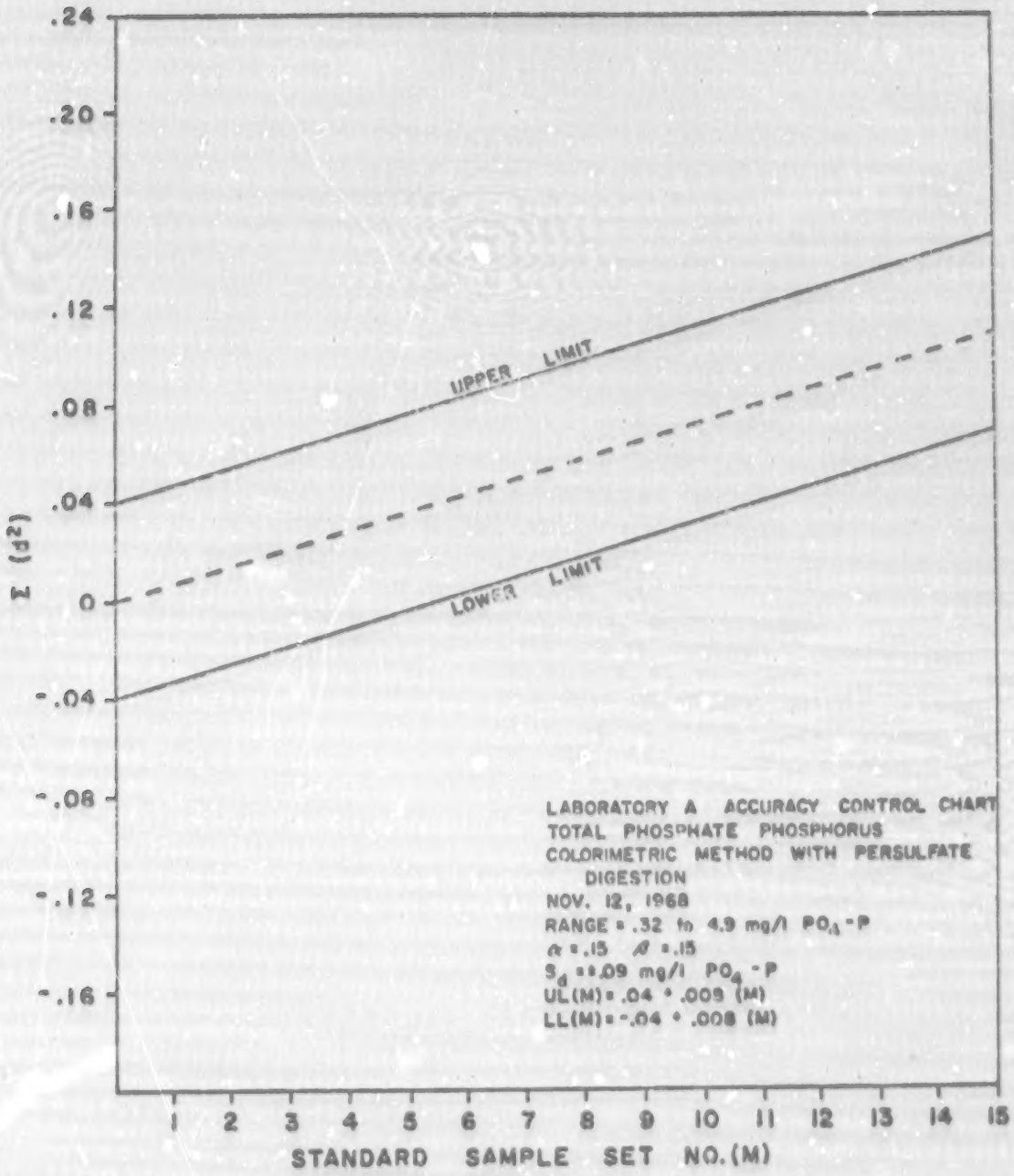

FIGURE 2 - ACCURACY CONTROL CHART

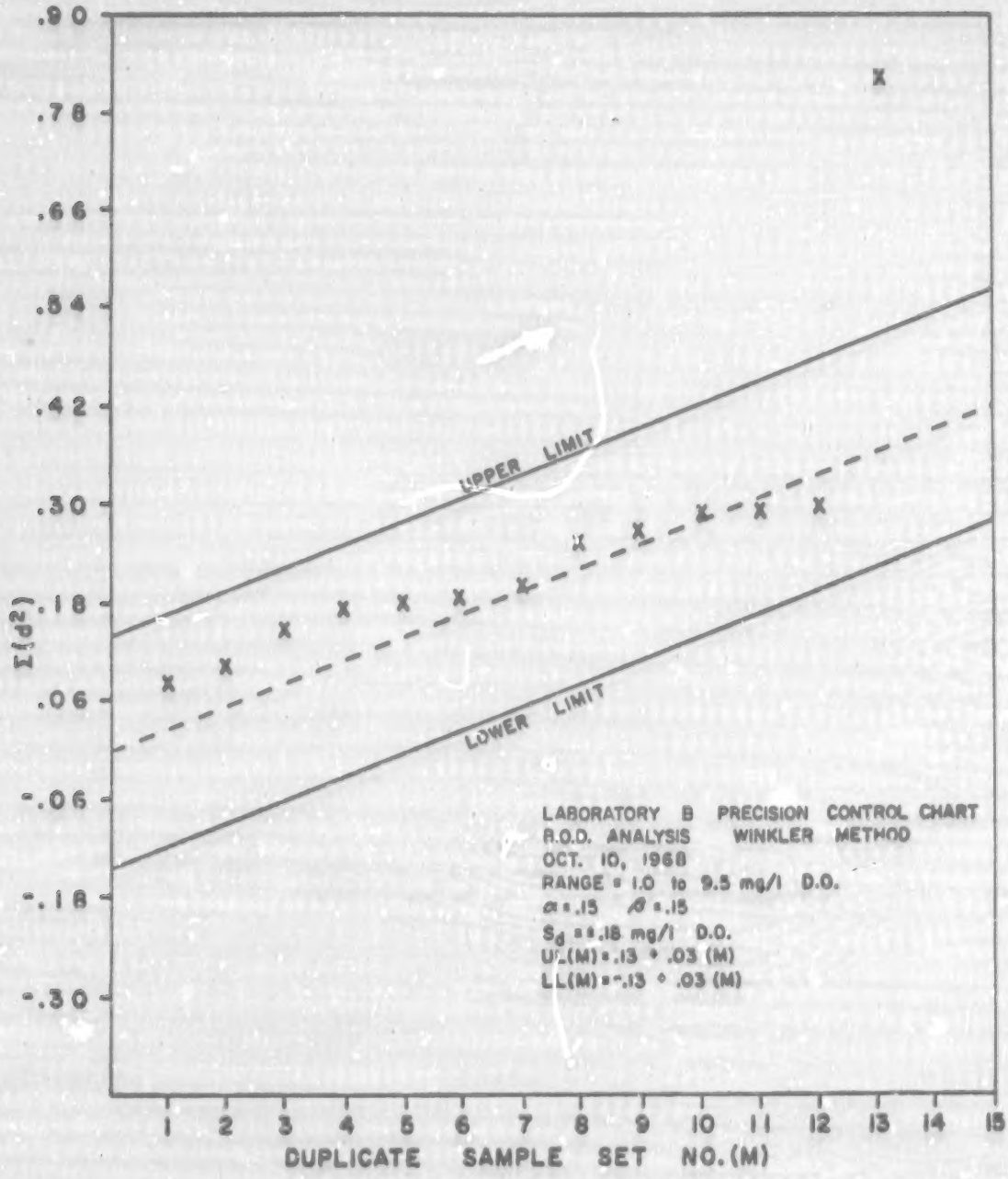

FIGURE 3 - OUT-OF-CONROL ON UPPER LIMIT

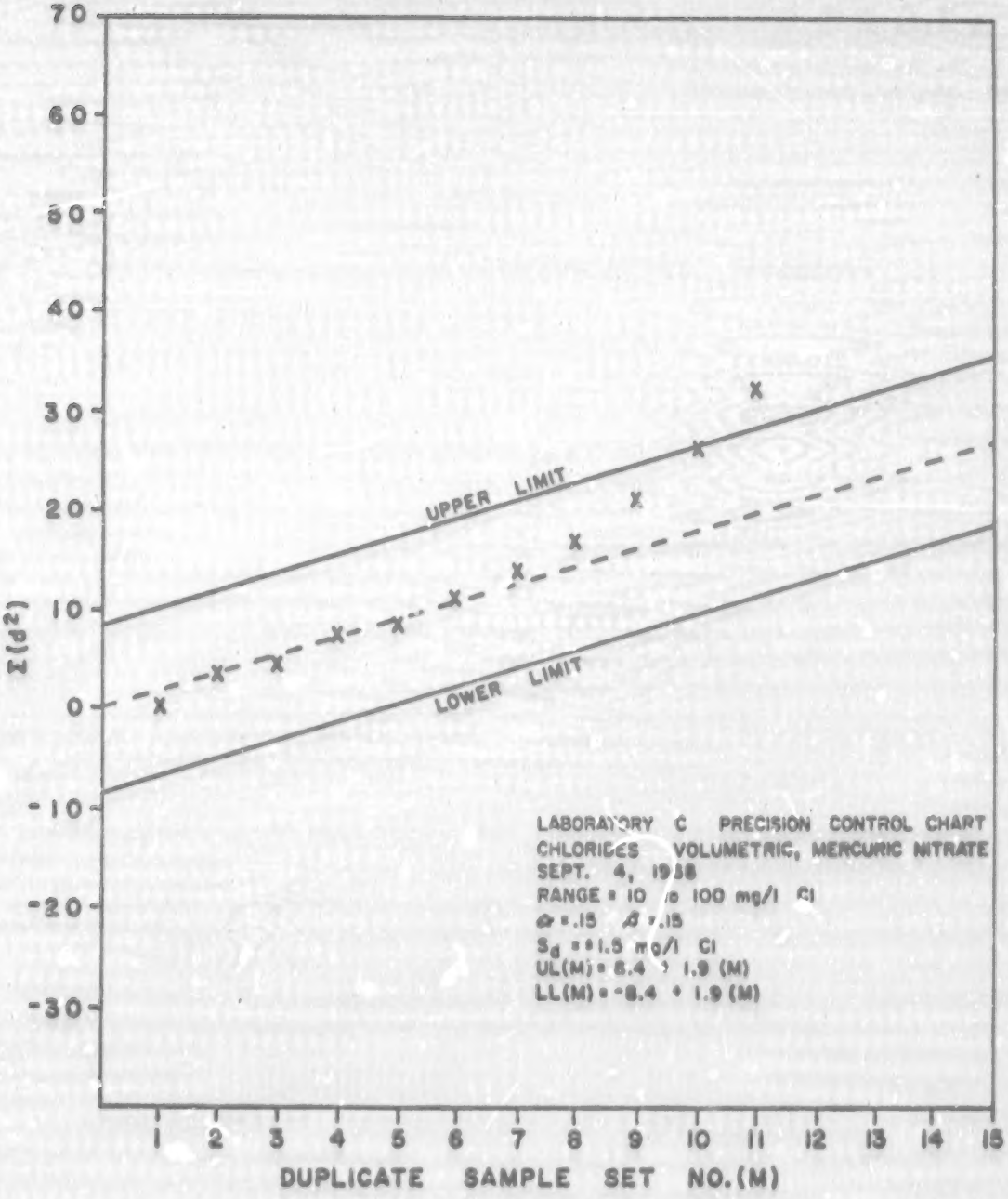

FIGURE 4 - OUT-OF-CONTROL ON UPPER LIMIT
(CONTINUOUS ERROR TREND)

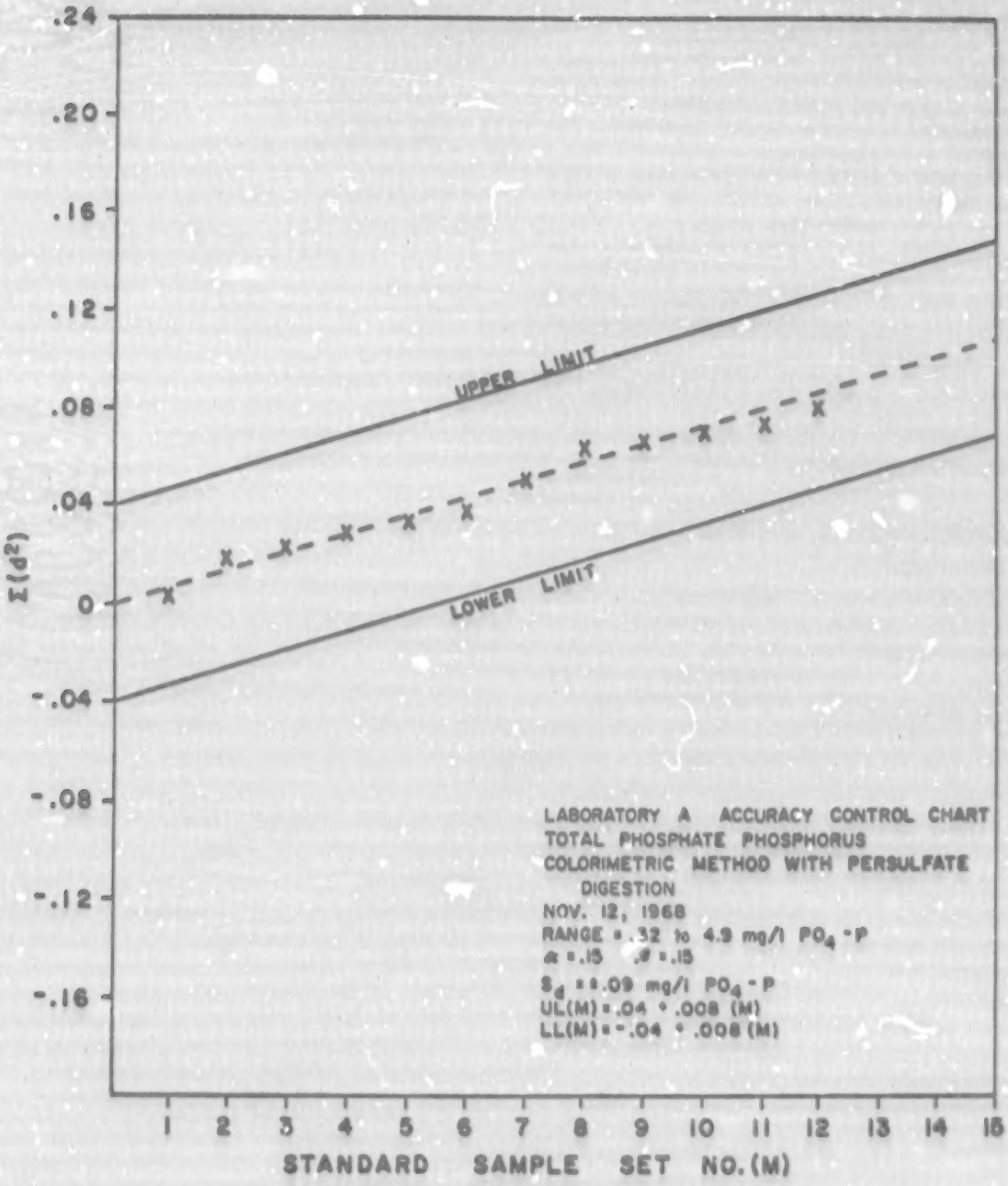

FIGURE 5 - IN - CONTROL WITHIN LIMITS

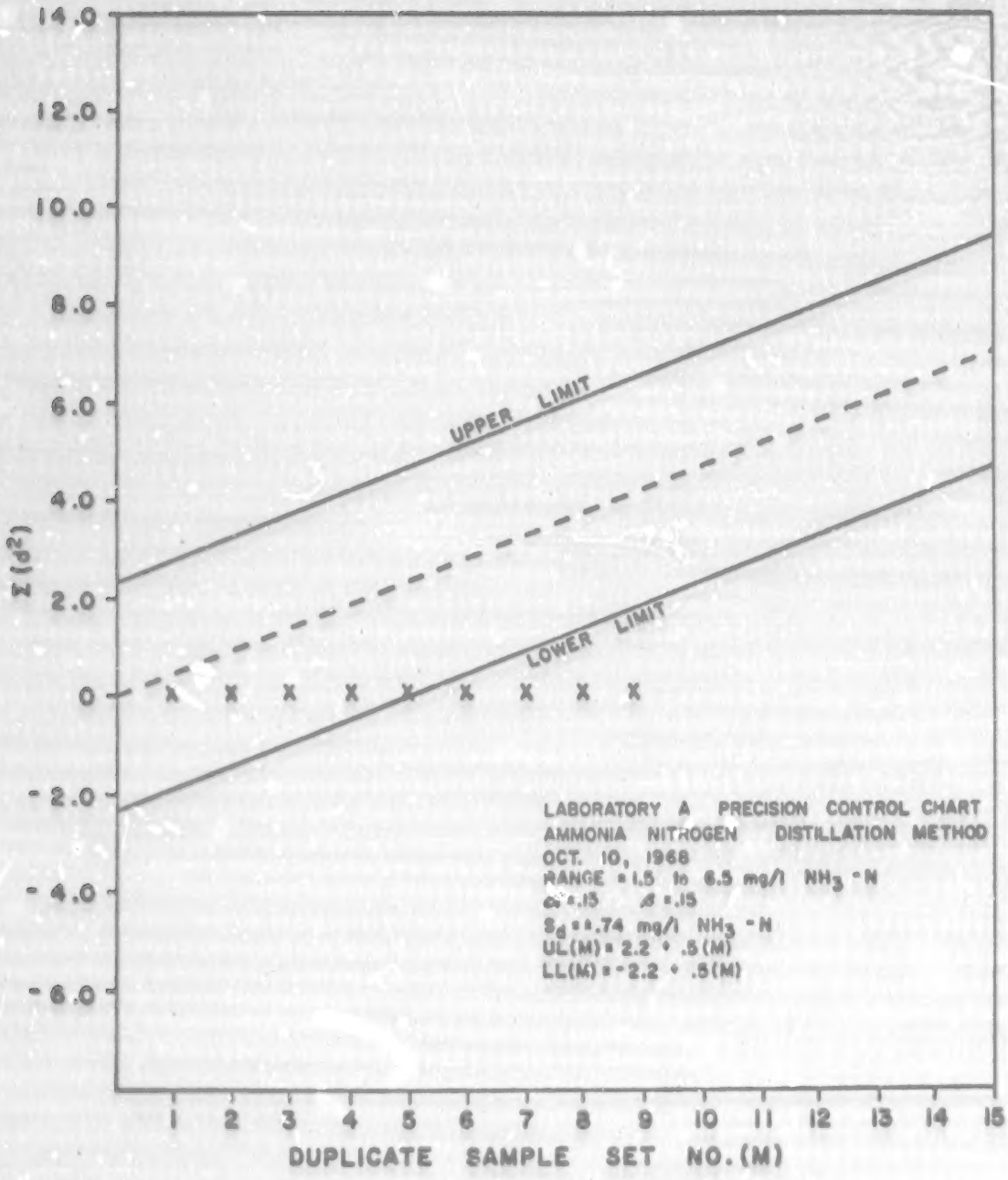

FIGURE 6 - OUT-OF-CONTROL ON LOWER LIMIT

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THE FEDERAL WATER POLLUTION CONTROL ADMINISTRATION'S ANALYTICAL QUALITY CONTROL PROGRAM*

by

Robert E. Crowe**

INTRODUCTION

The Federal Water Pollution Control Administration (FWPCA) gathers water quality data to determine compliance with water quality standards, to provide information for planning water resources development, to determine the effectiveness of pollution abatement procedures, and to assist in research and technical services activities. The sources of these data are not only the FWPCA laboratories but other Federal, city, State, and industry laboratories.

In a large measure the success of the pollution control program rests upon the reliability of the information provided by the data collection activities.

To insure the reliability of physical, chemical, and biological data, the FWPCA's Division of Research has established the Analytical Quality Control (AQC) Laboratory at 1014 Broadway, Cincinnati, Ohio. The program

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conducted by this laboratory is designed to assure the validity and, where necessary, the legal defensibility of all water quality information collected.

HISTORY

FWPCA recognized the need for an analytical quality control program in September 1966. Following this recognition, the first meeting on analytical quality control in the FWPCA was held in Cincinnati, Ohio, in January 1967. This meeting was attended by appropriate representatives from FWPCA's nine regions.

The purpose was to bring together as a working group those FWPCA personnel professionally and technically oriented and most knowledgeable in analytical chemical methods and procedures used to identify, measure, and characterize various types of water pollution. This group was designated as the Committee on Methods Validation and Analytical Quality Control. The Committee includes scientists who actively participate in the preparation of *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association (APHA) and in subcommittee and task group activities on Committee D-19 of the American Society for Testing and Material (ASTM). In addition one of the scientists is General Referee for Water, Subcommittee D of the Association of Official Analytical Chemists.

At the Cincinnati meeting subcommittees were chosen to study the then existing analytical methods for investigating water quality and to recommend the best of these methods for official designation by the FWPCA. These subcommittees were further broken down into specific parameter groups which, for one reason or another, were related. A Chairman was appointed for each of the subcommittees. The objectives set by these subcommittees to be accomplished by the fall of 1967 were:

1. To select those parameters which would be of use in the examination of water quality.
2. To review the analytical methods available for analyzing these parameters.
3. To formulate a list of the best methods available for immediate use.

Meeting these objectives represented the first major task to be accomplished by the newly organized Analytical Quality Control Section, which at the time was a part of FWPCA's Division of Pollution Surveillance.

In October 1967, the Committee on Methods Validation and Analytical Quality Control held its second meeting -- again in Cincinnati. At this time the initial task of the Analytical Quality Control Program was complete. The product of this first pioneering step was the publication and distribution of the *FWPCA Official Interim Methods for Chemical Analyses of Surface Waters*, September 1968.

In addition to the methods selection and validation activity, the Analytical Quality Control Program was being re-organized as a Laboratory of the FWPCA's Division of Research with the establishment of regional coordinators throughout the United States to coordinate program activities in each FWPCA region. Bringing the program to this point was a major step.

The AQC Laboratory is currently engaged in: (1) the selection and validation of methods for biological and microbiological determinations much the same as was done for chemical determinations; (2) intralaboratory quality control programs; (3) interlaboratory quality control programs; and (4) publishing the second edition of the *FWPCA Official Interim Methods for Chemical Analyses of Surface Waters*.

ORGANIZATION

The Analytical Quality Control Program of the FWPCA is carried out through an Analytical Quality Control Laboratory assisted by advisory committees on methods selection, regional quality control coordinators, and laboratory quality control officers. The organization and functions of these groups are described below.

Analytical Quality Control Laboratory

The Analytical Quality Control Laboratory is composed of five sections - Chemistry, Biology, Microbiology, Instrument Development, and Methods and Performance Evaluation. The laboratory staff coordinates the AQC Program, carries out methods development, conducts a continuing

reference sample service, and statistically evaluates laboratory performance.

Advisory Committee on Methods Selection

The Committee is composed of members appointed by FWPCA's Assistant Commissioner for Research and Development. These individuals may be selected from FWPCA programs or from outside agencies and are chosen because of their expertise in specific areas of analysis. The Committee's primary functions are to continually review the current literature and advise as to the selection of analytical methods for evaluation and certification. The director of the Analytical Quality Control Laboratory, acts as executive secretary to the Committee and coordinates its activities.

Regional Coordinators

To emphasize quality control participation at the regional level, each Regional Director appoints a coordinator whose primary function is to implement the analytical quality control program in all FWPCA laboratory activities within his region, and to assist or offer advice to appropriate groups outside the FWPCA concerning any phase of analytical quality control in the laboratory. Through individual quality control officers he provides leadership within the regional laboratory components, insuring the usefulness of this data for all regional functions. In addition, the coordinator keeps the Regional Director advised on analytical quality control activities in the laboratory under his

jurisdiction and inform the Analytical Quality Control Laboratory of his region's needs in methods development and data validation. The regional AQC coordinators and their respective regions, as designated in September 1968, are:

Northeast

Richard T. Dewling
North Atlantic Water
Quality Management Center
Federal Water Pollution Control
Administration
U. S. Department of the Interior
Edison, New Jersey 08817
FTS: 201-846-4611

Missouri Basin

Carl M. Walter
Missouri Basin Region
Federal Water Pollution Control
Administration
U. S. Department of the Interior
911 Walnut Street, Room 702
Kansas City, Missouri 64106
FTS: 816-374-3777

Middle Atlantic

Donald W. Lear
Laboratory Unit
Chesapeake Bay-Susquehanna
River Basin Project
Federal Water Pollution Control
Administration
U. S. Department of the Interior
Annapolis Science Center
Annapolis, Maryland 21400
FTS: 301-597-3311
Ask for: 268-5038

South Central

Robert E. Crowe
Robert S. Kerr Water
Research Center
Federal Water Pollution Control
Administration
U. S. Department of the Interior
P. O. Box 1198
Ada, Oklahoma 74820
FTS: 405-236-2311
Ask for: 332-8800 (ext. 235)

Southeast

William T. Donaldson
Southeast Water Laboratory
Federal Water Pollution Control
Administration
U. S. Department of the Interior
College Station Road
Athens, Georgia 30601
FTS: 404-548-7211
7266

Southwest

John C. Merrell, Jr.
Southwest Region
Federal Water Pollution Control
Administration
U. S. Department of the Interior
Phelan Building
760 Market Street
San Francisco, California 94102
FTS: 415-556-5876

Ohio Basin

Robert L. Booth
Analytical Quality Control Laboratory
Division of Water Quality Research
Federal Water Pollution Control Administration
U. S. Department of the Interior
1014 Broadway
Cincinnati, Ohio 45202
FTS: 513-684-2900

Northwest

Daniel F. Krawczyk
Pacific Northwest Water Laboratory
Federal Water Pollution Control Administration
U. S. Department of the Interior
200 South 35th Street
Corvallis, Oregon 97330
FTS: 503-752-4339

Great Lakes

Clifford Risley, Jr.
Great Lakes Region
Federal Water Pollution Control Administration
U. S. Department of the Interior
33 East Congress Parkway, Room 410
Chicago, Illinois 60605
FTS: 312-353-5638

Laboratory Quality Control Officers

This officer, usually a senior member of the laboratory staff, is appointed by the Laboratory Director and is responsible, through him, to the Regional Quality Control Coordinator. He is concerned with the analytical quality control of FWPCA laboratories within the region.

RESPONSIBILITIES

National

The national responsibilities of the FWPCA's analytical quality control program are primarily those of the Analytical Quality Control

Laboratory in Cincinnati, Ohio. These responsibilities are described below.

Methods Research

Although analytical methods are available for most of the routine measurements used in water pollution control, there is a continuing need for improvement in sensitivity, precision, accuracy, and speed. Development is required to take advantage of modern instrumentation in the water laboratory. In microbiology, the use of new bacterial indicators of pollution, including pathogens, creates a need for rapid identification and counting procedures. Biological collection methods need to be standardized to permit efficient interchange of data. The Analytical Quality Control Laboratory devotes its research efforts to the improvement of the routine tools of the trade; therefore, it has nationwide responsibility for the guidance of a program to develop reliable analytical methods for water and wastewater analyses.

Methods Selection

Assisted by the advisory committee, the AQC Laboratory provides the program for the selection of the best available procedures in water and waste analyses. This includes certification of the methods through an adequate testing program. Through the publishing of FWPCA methods manuals, updated regularly, the program insures the uniform application of analytical methods in all laboratories of the FWPCA.

Interlaboratory Quality Control

The Analytical Quality Control Laboratory is responsible for maintaining a reference sample program for methods verification and laboratory performance evaluation of all FWPCA laboratories. This also includes the validation of chosen procedures of existing or new developmental methods of analysis.

Intralaboratory Quality Control

To maintain a high performance level in daily activities, every analytical laboratory must utilize a system of checks on the accuracy and precision of reported results. While this is a part of the responsibility of the analyst and his supervisor, the Analytical Quality Control Laboratory is responsible for guidance in the development of model quality control programs which can be incorporated into the laboratory routine.

Regional

The regional responsibilities are essentially those of the regional coordinator and the laboratory quality control officer of a particular region. The regional coordinator is responsible for implementing the nationwide program in the FWPCA regional laboratory and maintaining appropriate relations with other federal agencies, with state and interstate pollution control agencies, and with industry to encourage their use of the FWPCA methods and their participation in the analytical quality

control effort. In addition, the regional coordinator is responsible for bringing to the attention of the AQC Laboratory any special needs of his region in analytical methodology and any analytical quality control problems that occur.

The laboratory quality control officer is responsible for carrying out an intralaboratory quality control program among the FWPCA laboratories in his region, assuring the use of certified methods by the laboratory staffs and securing participation in regular check sample analyses.

FWPCA OFFICIAL METHODS

Missions assigned to FWPCA by the Water Quality Act of 1965 and the Clean Water Restoration Act of 1966 created a need for methods capable of developing water quality data to measure the effectiveness of the Nation's water pollution control programs. The methods must be uniform throughout the Administration and based on sound, scientific investigations. Further, they must be available to all other elements of the water pollution control field involved in, or affected by, water quality standards, and must be acceptable as legally defensible in Federal and State enforcement actions to abate water pollution.

The first edition of the *FWPCA Official Interim Methods for Chemical Analyses of Surface Waters* was a major step in this direction and the first product of the FWPCA's Analytical Quality Control Program.

To acquaint you who are attending this conference with the manual and its implications, it seems appropriate to discuss it at this time. As I have said, this manual represents the selections made by a committee of senior FWPCA laboratory personnel, working under the guidance of the Analytical Quality Control Laboratory. The Committee consulted all the available literature, including *Standard Methods for the Examination of Water and Wastewater*, *ASTM Manual of Industrial Water*, and current technical journals.

This manual in first edition was limited in number, and thus is not available to the general public. However, the second edition, now being compiled, will be available for all who wish copies in July 1969. Several copies of the first edition will now be passed among you for your rapid perusal.

This manual, in second edition can be obtained after July 1, 1969 by sending a request to your regional coordinator. For those of you living in this region (South Central) the address is

Robert S. Kerr Water Research Center
Federal Water Pollution Control Administration
U. S. Department of the Interior
P. O. Box 1198
Ada, Oklahoma 74820
Attention: Robert E. Crowe, AQC Regional Coordinator.

SUMMARY

In order that industry, state and interstate pollution control agencies, the FWPCA and other Federal agencies can effectively relate

water quality data and feel secure that water quality data produced from all the laboratories concerned are valid, the FWPCA organized an analytical quality control program. The organization and responsibilities of this program nationally and regionally have been discussed.

The success of this program depends upon the active participation of managers, supervisors, and analysts in not only the FWPCA, but in all groups concerned with characterizing and maintaining water quality. Therefore, it is essential that the philosophy of quality control be understood and accepted by all levels in any laboratory organization.